Thomas Scientific

# Analytical workflows in cannabis testing

**Supelco Product offerings for your customers**

Kathy Stenerson, Analytical Sciences Liaison

Distributor Training, Sept. 10, 2021

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# agenda

01 Why test cannabis?

02 Regulatory landscape

03 Workflow overviews

04 Technology highlights

05 Collateral



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# Why Test Cannabis?

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# What is Cannabis?

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**Three species**

*Cannabis sativa*

*Cannabis indica*

*Cannabis ruderalis*

**Usage**

Medicinal

Industrial

Recreational

**Qualifying Condition**

- Cancer
- Glaucoma
- HIV/AIDS
- Multiple sclerosis
- Epilepsy
- PTSD

**Commercial Formats**

Plant material

Oils/Extracts

Edibles

Creams

Pharmaceuticals




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# Why Test Cannabis?

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## Patient & Consumer Health

**Purity**
- Safety
- Impurities

**Pathogens, pesticides, heavy metals, residual solvents, fungal toxin**

**Potency**
- Label Claim
- Correct dosage?
- Correct Strain?
- Hemp vs Cannabis

**Cannabinoids, terpenes**

## Optimize Production

**Agriculture & Genetics**
- Sex identification
- Strain integrity
- Selective breeding
- Cannabinoid profile

**Cannabinoids, Pesticides**

**Potency**
- Timing for harvesting
- R&D, QC of strain characteristics
- Processing

**Cannabinoids, terpenes**

* Terpenes are the aromatic compounds in Cannabis that are believed to have an important influence on the effects of cannabis through what is known as the "entourage effect". They are also crucial to strain identification.



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# Regulatory Landscape

# 02

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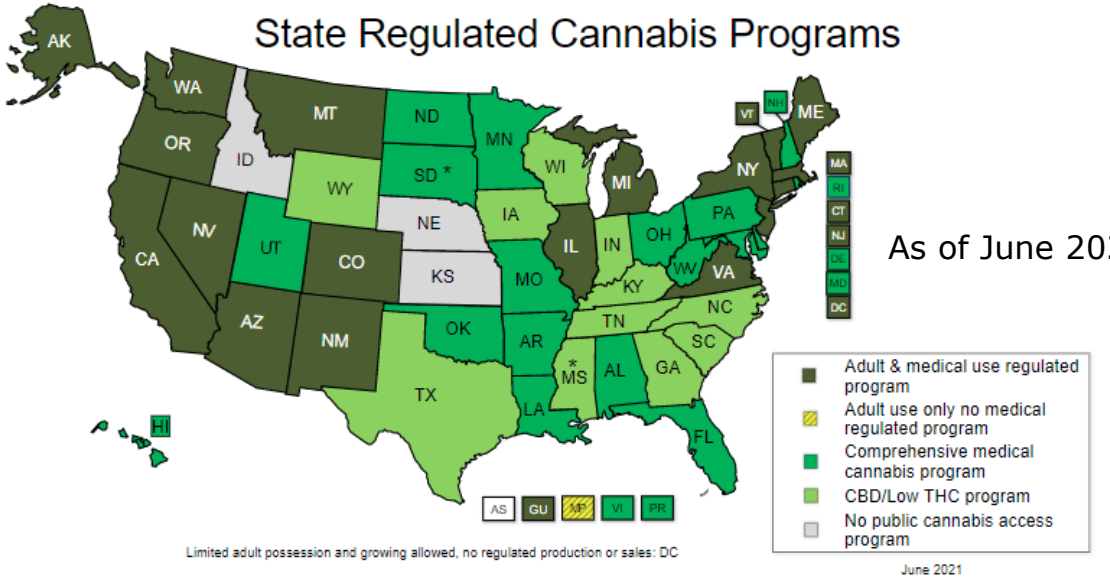
# Current Legal Environment

Highly fragmented legal and regulatory environment globally

**Canada** –
Legal both for medical and recreational use




As of June 2021

*ncsl.org*

| United States | |
|---|---|
| Category | # States Legal |
| Medical & Recreational Marijuana | 18 States, + Washington DC |
| Medical Marijuana | 18 States |
| CBD/Low THC (hemp) | Federally legal |

# Regulatory Landscape

**Cannabis still listed under Schedule 1 of the Controlled Substance Act**

- reserved for drugs that have "no currently accepted medical use"
- Under jurisdiction of DEA (DoJ)

**No national regulatory policy in place for the Cannabis industry**

- Quality control
- Safety

**Possession of Cannabis has been decriminilized by many states & municipalities**

# Potency

## Regulated Cannabis testing

### Cannabinoids

- Major therapeutic and psychoactive chemical component of cannabis
- >100 cannabinoids present, mainly in extremely minute amounts
- State regulations range from 3– 6 different compounds, most states require THC, CBD and CBN (cannabinol)


### Optional: Terpenes

- Strong aromatic component to Cannabis
- Potential for synergistic effects with cannabinoids – "Entourage effect"
- Useful in strain identification

# Purity

## Regulated Cannabis testing

Impurities are further concentrated in the extraction process

### Pesticides

- Many states have developed own lists (Ex: OR = 47, NV = 33 required)
- Fungicides are particularly important

### Heavy Metals

- Uptake of trace levels of heavy metals such as arsenic, cadmium, mercury, and lead from soil and through the use of certain fertilizers
- Some states following United States Pharmacopeia (USP) <232> for elemental impurities

### Filth/Foreign Materials

- Hair, insects, feces, packaging contaminants, and manufacturing waste and by-products

### Residual Solvents

- Extracting solvents and processing chemicals left over from cannabinoid extraction process (butane is quite common)

### Microbiology Impurities

- Morphology of medical cannabis makes it especially vulnerable to fungal infections
- Microorganisms can be introduced through handling, transportation and processing

### Moisture Content & Water Activity

- Evaluated in dried plant material, various techniques applied

# California cannabis regulations

Spotlight on CA required testing

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California Bureau of Medical Cannabis Regulation

## 6 Cannabinoids

- THC
- THCA
- CBD
- CBDA
- CBG
- CBN

## 22 Residual Solvents and Processing Chemicals

## 66 Residual Pesticides

## 4 Heavy Metals

- Cadmium, Lead, Arsenic, Mercury

## Mycotoxins

- Aflatoxins B1, B2, G1, G2 plus Ochratoxin

## Microbiological Impurities

- E-coli, salmonella, aspergillis

## Moisture Content & Water Activity

## Filth/Foreign Materials

Optional: **Terpenes**

**Certificate of Analysis**



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# Pennsylvania cannabis regulations

Spotlight on PA required testing


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### Cannabinoids

- THC
- CBD
- THCA
- CBDA
- CBG
- CBN

### Terpenes

- No specific list

### 2 Residual Solvents and Processing Chemicals

- Butane, Ethanol

### 62 Residual Pesticides

### 4 Heavy Metals

- Cadmium, Lead, Arsenic, Mercury

### Mycotoxins

- Aflatoxins B1, B2, G1, G2 plus Ochratoxin

### Microbiological Impurities

### Moisture Content & Water Activity

### Filth/Foreign Materials



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# Types of customers

## 3 Government

USDA

CDC

FDA

Steep Hill

## 4 Testing Labs

sclabs™

CW ANALYTICAL
SAFETY. QUALITY. SCIENCE.

TECHNION
Israel Institute of Technology

## 2 Cannabis Product Manufacturers

LenitivLabs
SAFE + SOOTHING + SCIENCE

ELIXINOL™

## 5 Research Institutions

THE UNIVERSITY of MISSISSIPPI

CMCR
Center for Medicinal Cannabis Research

## 1 Growers with in-house QC

America's Cannabis Brand
GFarmaLabs™
a G FarmaBrands Company

CHALICE FARMS™

## 6 Pharma

GW pharmaceuticals

Arena
Arena Pharmaceuticals

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# workflow overviews

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# What is an analytical workflow?

## LC, LC-MS, GC, GC/MS

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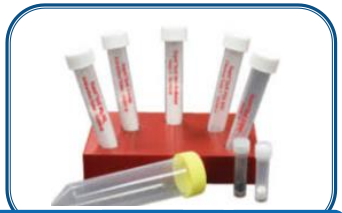
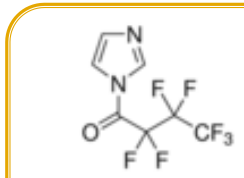
**Sample Collection**

- SPME
- Sample collection tubes
- Headspace vials



**Sample Isolation & Preparation**

- QuEChERS
- SPE Cartridges & Accessories
- Syringe filters
- Reference Material




**Derivatization (optional)**

- GC Reagents
- LC Reagents

**Analysis**

- GC columns
- LC Columns
- Accessories
- Reference Material

Lab Water

Life Science Chemicals

Lab Ware



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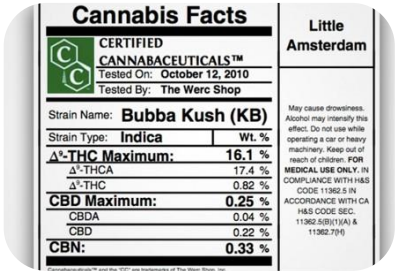
# Main Analytical Workflows in Cannabis Testing Labs




Cannabinoids

- THC, CBD, etc.


Terpenes

- Limonene, pinene, etc.

Residual solvents

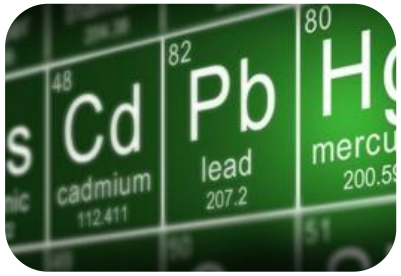
- Butane, isobutane, ethanol, etc.

Pesticides

- Insecticides, herbicides, anti-fungals

Mycotoxins

- Aflatoxins, Ochratoxin



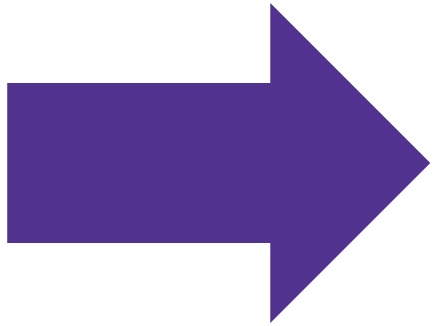

Heavy Metals

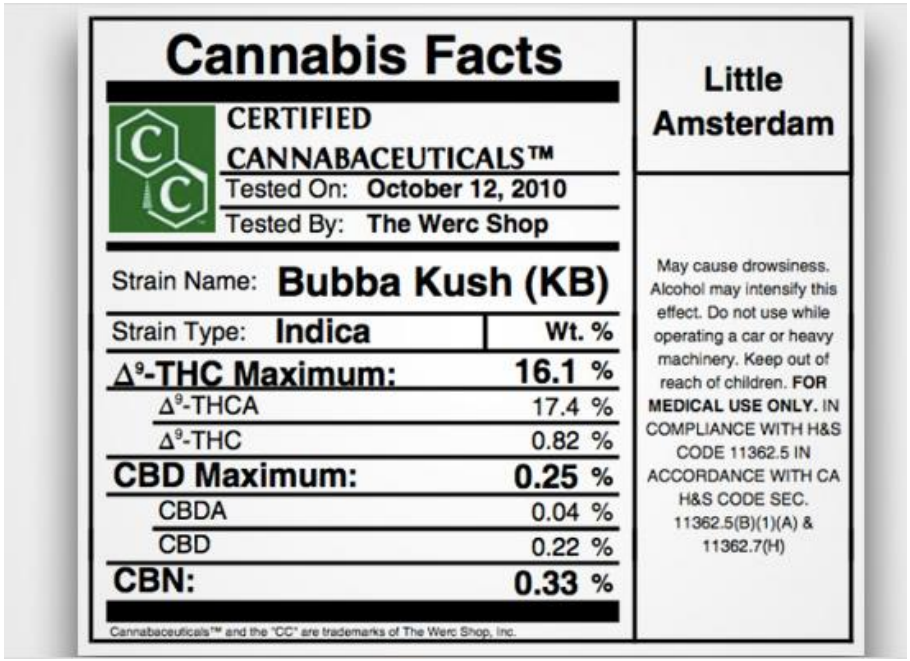
- Cd, As, Pb, Hg

Microbiology

- Mold, yeast, bacteria

# Cannabinoids






Most common test –required by all states!

# Typical Cannabinoid Testing Workflow

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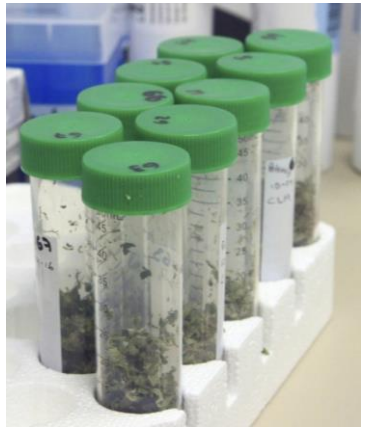
Homogenize & weigh sample → Solvent Extraction → Dilution → Filter → HPLC analysis



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# Cannabinoids

## Identify and Quantitate Cannabinoids

**MilliporeSigma Products:**

Sample prep

Solvents

- Ethanol, methanol commonly used

Sample vials

Syringe filters

Analysis

HPLC columns

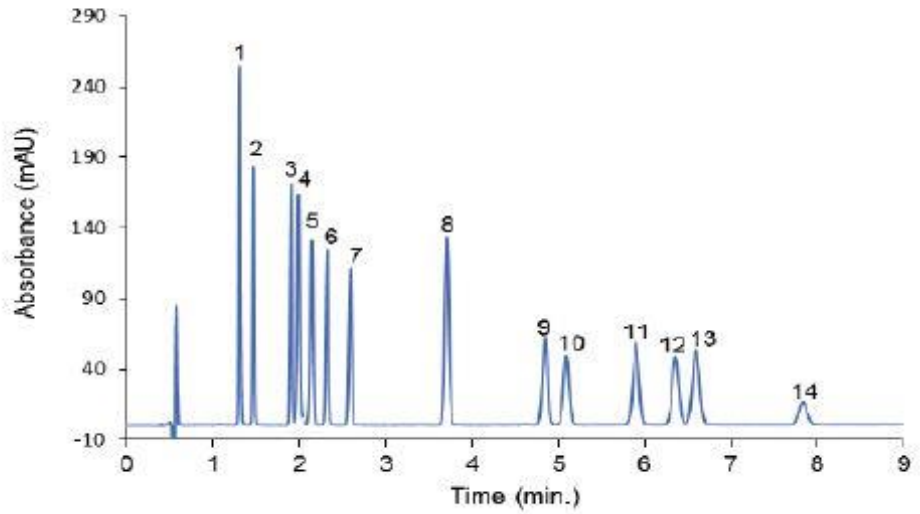
- Ascentis® Express C18
- Chromolith®

HPLC grade solvents for mobile phase

- Acetonitrile, methanol, water - LiChrosolv® & OmniSolv®

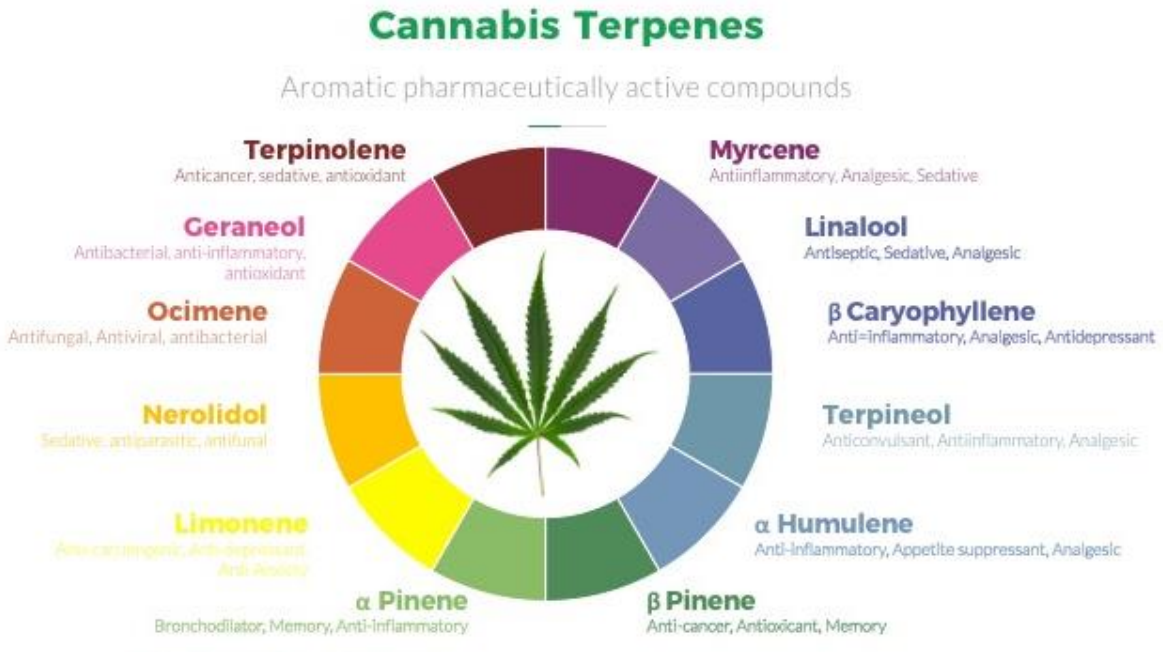
Reference materials

- Cerilliant cannabinioid CRMs (individuals and mixes)

# Identify and Quantitate Terpenes

Terpenes confer the fragrance of Cannabis and can have pharmacological effects (independent of or possibly in conjunction with, cannabinoids).


Two typical analysis workflows

1. Headspace GC analysis
2. Solvent extraction, GC analysis

# Identify and Quantitate Terpenes

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**MilliporeSigma products for sample preparation:**

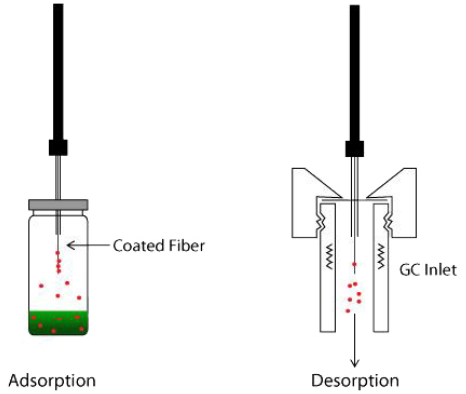
Headspace Extraction

- Autosampler Vials & Caps
- SupraSolv® Headspace grade solvents
- SPME

OR

Solvent Extraction

- Vials – for extraction and autosampler
- OmniSolv ® & SupraSolv ® GC grade solvents







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# Identify and Quantitate Terpenes

## Products for Analysis:

Standards

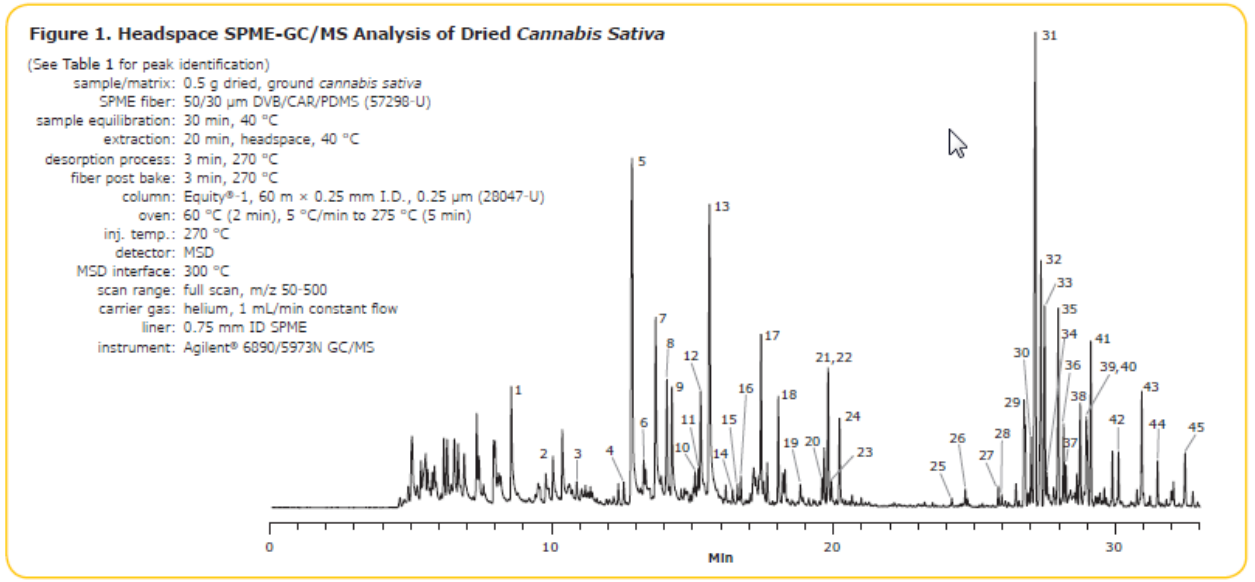
- Terpene mixes
- Individual terpenes

GC Columns

- Equity® -1, SLB® -1ms
- SLB® -5ms

GC accessories

- Inlet consumables
- Gas purification & handling



# Typical Pesticide Testing Workflow

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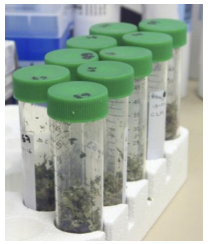
Homogenize & weigh sample → Extraction → Cleanup → Solvent exchange and/or dilution → Filter → GC-MS/MS & LC-MS/MS analysis





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Pesticides

# Identify and Quantitate Pesticide Residues


**MilliporeSigma products for sample prep and analysis**

QuEChERS supplies

- Salts (for extraction step)
- Sorbents (for cleanup step)

SPE

Solvents

- For extraction (methanol, acetonitrile, water)
- For MS analysis (LiChrosolv® & Omnisolv®)

Reference Materials

Labware

GC and HPLC columns

- Ascentis Express® HPLC Columns for LC-MS/MS
- SLB® -5ms column for GC-MS/MS

Instrument accessories & consumables – GC and HPLC

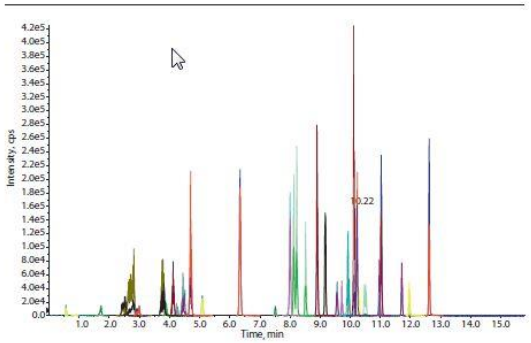



**Figure 3** LC/MS/MS chromatogram of spiked group of 29 compounds used in this validation on Ascentis Express RP-Amide

# Residual Solvents

## Identify and Quantitate Residual Solvents

Solvents are used in process scale extraction of Cannabis

# Residual Solvents

## Identify and Quantitate Residual Solvents

**MilliporeSigma Products for Residual Solvents Analysis:**

SupraSolv® Headspace grade solvents

Headspace vials & caps (to fit autosampler)

SPME

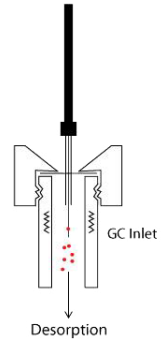
Reference materials

- USP class I, II, III solvent mixes

GC Columns

- SPB® -624
- Vocol®

Adsorption




Desorption

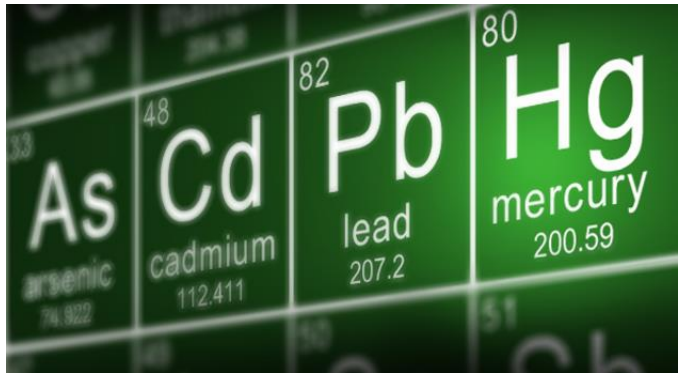
**Typical Workflow**

Weigh sample → Dissolve in solvent → Analyze by HS-GC

# Metals

## Identify and Quantitate Heavy Metals

Heavy metals are in soil and water; arise from atmospheric aerosols, or constituents of fertilizers, pesticides, herbicides, and fungicides; likely toxic; bio-accumulate in Cannabis




# Metals

## Identify and Quantitate Heavy Metals

**MilliporeSigma products for sample prep & analysis**

High purity reagents for digestion & dilution

- Nitric and Hydrochloric Acids – Suprapur®, Ultrapur, EMSURE
- Hydrogen Peroxide - Suprapur®, Ultrapur
- Water – Ultrapur bottled, ultrapure Milli-Q

Certified Reference materials for ICP-MS analysis

- Single element solutions
- Mixes



Typical metals workflow

Grind & weigh sample → Digestion → Dilution into high purity water → ICP-MS analysis

# Mycotoxins

## Identify and Quantitate Mycotoxins

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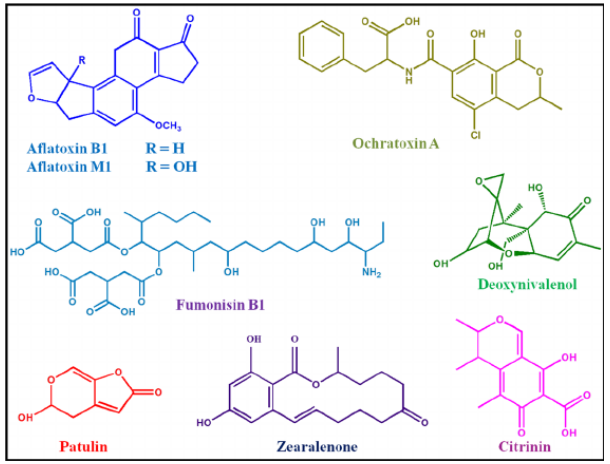
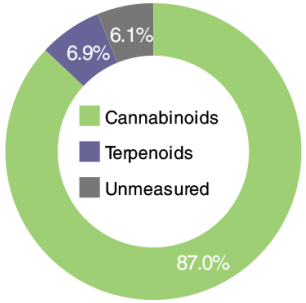
Mycotoxins are natural defense compounds of fungi. Aflatoxins may be present on Cannabis.




Some labs analyze mycotoxins together with pesticides

**Sample Overview**


**Sample Details**

| | |
|---|---|
| Mycotoxin | Not Detected |
| Pesticide | Not Detected |

For more information about this report, including how to calculate your own approximate post-decarboxylate THC and CBD values, please visit www.steephilllab.com/FAQ.

Grind & weigh sample → Solvent Extraction → Cleanup → LC-MS/MS analysis

# Mycotoxins

## Identify and Quantitate Mycotoxins

**MilliporeSigma Products for Sample Prep (mycotoxins only):**

Liquid extraction

- LiChrosolv® and OmniSolv ® high purity solvents

SPE - Supel™ Tox AflaZea

Reference Materials

- Individuals & mixes
- Isotopically labeled for use as internal standards


**Analysis**

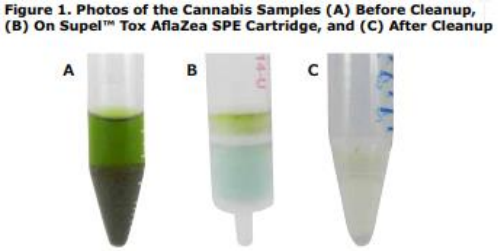



Figure 1. Photos of the Cannabis Samples (A) Before Cleanup, (B) On Supel™ Tox AflaZea SPE Cartridge, and (C) After Cleanup

# Mycotoxins

## Identify and Quantitate Mycotoxins

### MilliporeSigma Products for Analysis:

Reference Materials

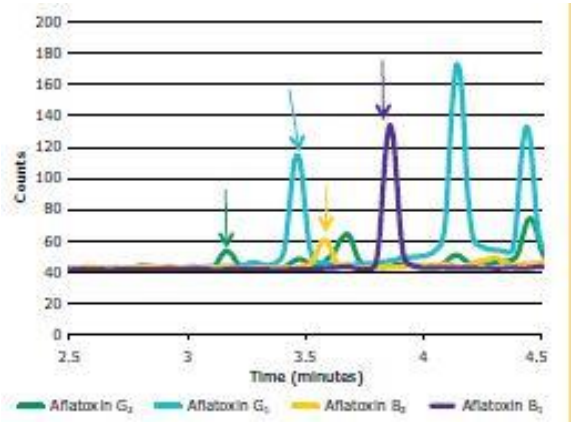
- ID
- Calibrant

HPLC Columns

- Ascentis® Express (many different chemistries)

LC/MS Grade Solvents

- LiChrosolv®
- Omnisolv®



# Microbiology
## Identify and Quantitate Microorganisms

Cannabis has antimicrobial properties, but microorganisms can be introduced throughout the handling, transporting and processing phase after harvest.

**Microbes of interest:**

Aspergillus (4 species)

STEC (Shiga toxin-producing E. Coli)

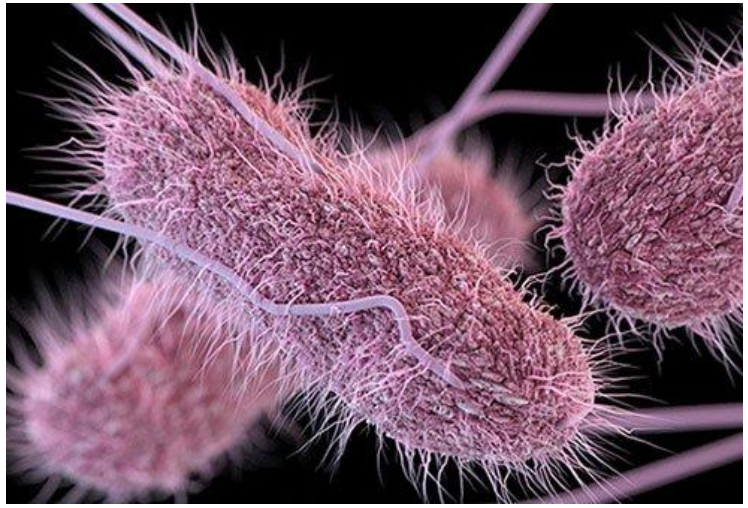
E. Coli

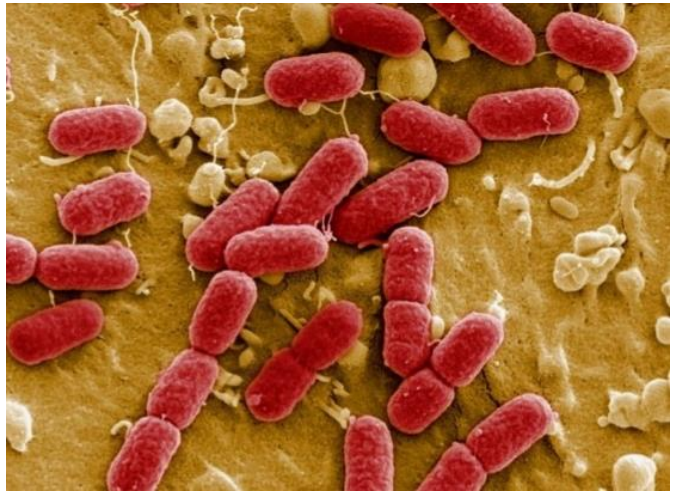
Salmonella

Total Yeast and Molds

Total Aerobic Counts

Total Coliform





# Microbiology

## Identify and Quantitate Microorganisms

### 1. Traditional Plate method

- Dehydrated media (solid granules or powder format)
- Ready to Use media (mostly liquid format; some solid)

### 2. MC Media pads

- Yeast & mold
- Total aerobic
- E. Coli & coliform


### 3. Assurance GDS – PCR

- Salmonella
- E-coli
- STEC (Shiga-toxin producing E-coli)

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# Technology Highlights

## (what Makes us different?)

04

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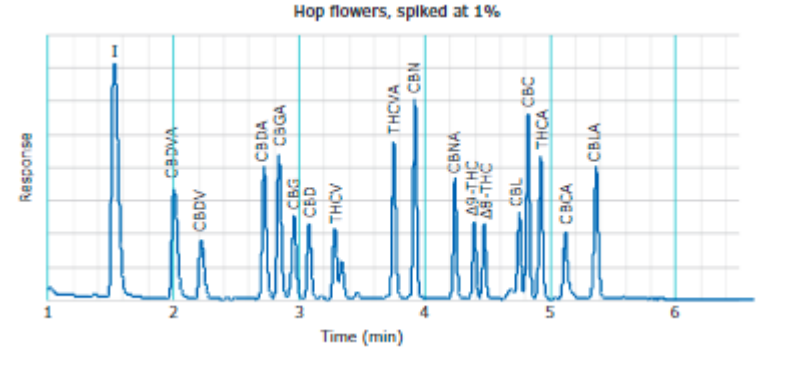
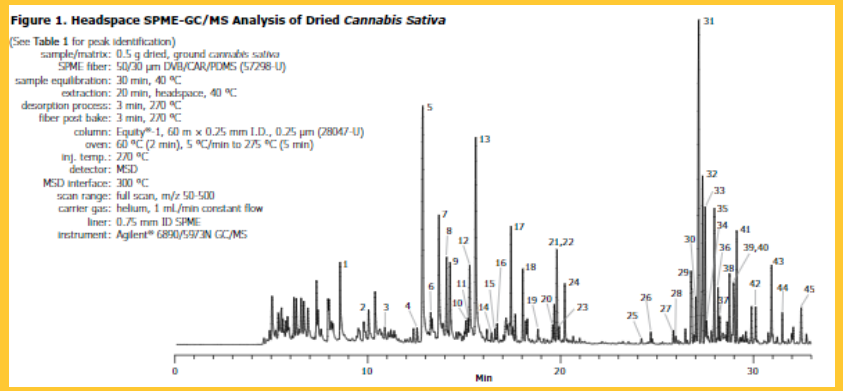
# Differentiating & Key Products




## Cannabinoid testing

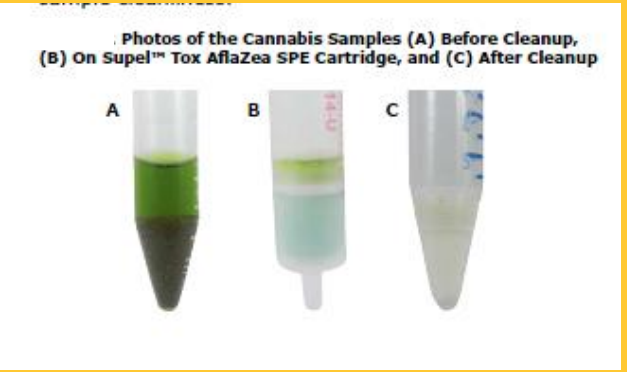
- Cerilliant® CRMs – trusted name, highest quality, years of experience
- Ascentis® Express HPLC columns – for fast, efficient and rugged separations
- Chromolith® HPLC columns – unique monolithic silica, matrix tolerant, rapid analysis




## Terpenes

- SLB® -5ms GC column – low bleed, MS grade
- SupraSolv® Headspace grade solvents – ultra clean, suitable for headspace
- SPME – solid phase microextraction; sensitive, reusable, easy to automate. We are the leaders in this.

# Differentiating & Key Products

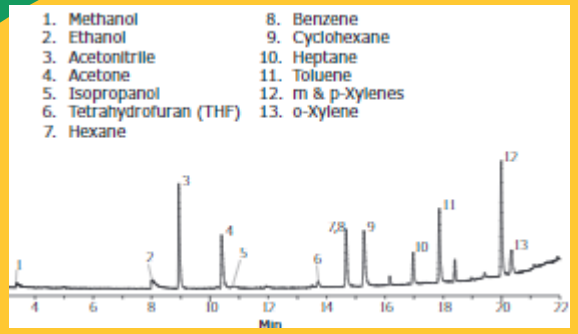

## Pesticides

- Ascentis® Express HPLC columns – highly efficient, fast, rugged analyses
- Supel™QuE Verde sorbent - for QuEChERS cleanup of green samples with improved recovery of planar pesticides
- Pestanal® Isotope labeled pesticides – for use as internal standards

## Mycotoxins

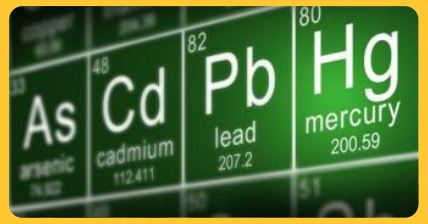
- Supel™ Tox AlfaZea – simple pass-through SPE cleanup for aflatoxins
- Ascentis® Express HPLC columns - highly efficient, fast, rugged analyses
- CRMs – individual mycotoxins and mixes, also isotopically labeled for use as internal standards.

# Differentiating & Key Products




## Residual Solvents

- Class I, II, III residual solvent CRMs
- Suprasolv® Headspace grade solvents
- SPME – a less expensive alternative to conventional headspace




## Heavy Metals

- CRMs – mixes and individuals
- Emsure, Ultrapure and Suprapur High Purity reagents – customer can choose based on their needs

## Microbiology

- Vitroids™ & Lenticule® Certified Reference Microorganisms – fast, reliable, easy to use
- MC Media Pads® - alternative to traditional plates, simple and easy to use
- Granulated and ready to use culture media

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# collateral

05

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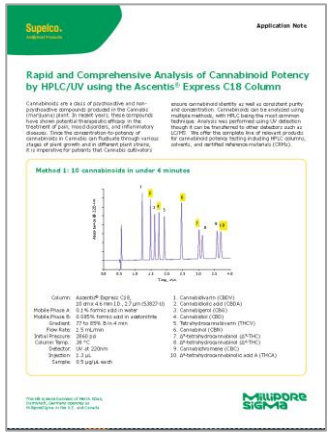
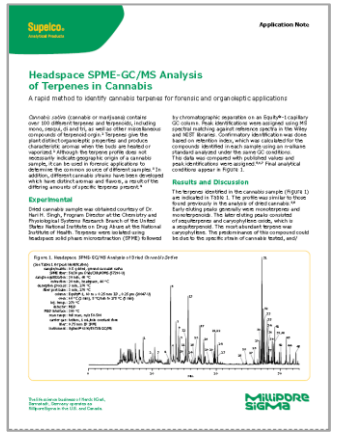
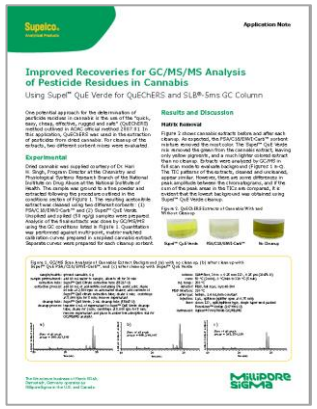
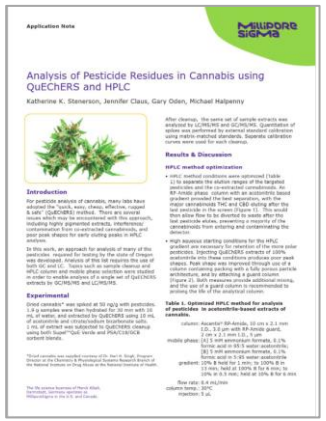
# Application Notes

- Cannabinoids
  - MS_AN6577EN
  - MS_AN2607EN
- Terpenes
  - MS_AN5192EN
- Pesticides
  - MS_AN5191EN
  - MS_AN1655EN
  - MS_AN7780EN
- Mycotoxins
  - MS_AN5190EN
- Heavy Metals

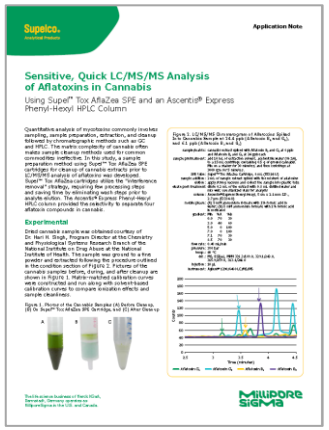



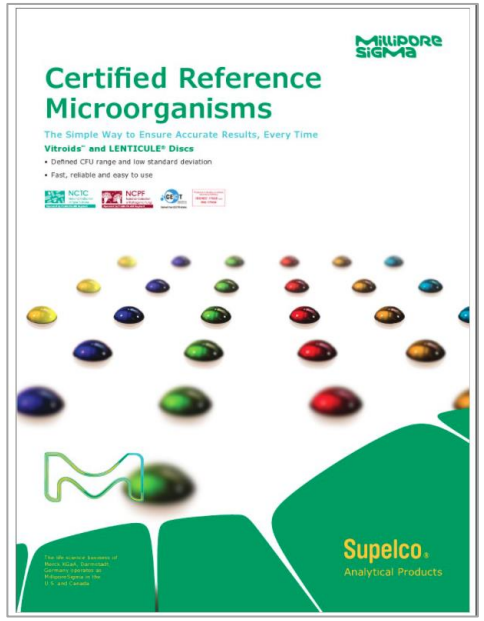



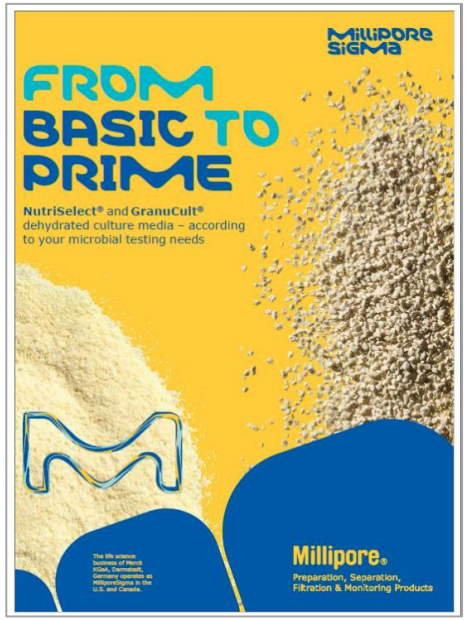




# Brochures

- Microbiology
  - MS_BR1756EN
  - MS_BR1710EN
  - MS_BR6639EN


More coming soon!!

# Website

Supelco®

## Analytical Testing

Beyond the Leaf


- NEW Workflow applications
- Product recommendations

Cannabis quality testing is mandated in all regions around the world where legalization took place. We offer the most comprehensive selection of analytical tools to promote safety and efficacy of cannabis products. From analytical sample prep, to high purity solvents, columns and certified reference materials, we offer solutions for your end-to-end cannabis testing workflow.

### ANALYTICAL CANNABIS TESTING SOLUTIONS

- Cannabinoids and Potency
- Terpenes
- Pesticides
- Mycotoxins
- Heavy Metals
- Residual Solvents
- Moisture Analysis

### RELATED TECHNICAL RESOURCES

As your partner in analytical testing, we've made step-by-step guides available for your cannabis workflow.

**Analysis of 17 Cannabinoids in Hemp and Cannabis**

HPLC separation of 17 important cannabinoids including CBD, delta 9 THC and THCA. Read the application note

**Complete Workflow for Comprehensive Cannabis Terpenes Analysis**

Complete workflow for the comprehensive analysis of terpenes in cannabis

**ICP-MS Analysis of Heavy Metals in Cannabis Sativa**

Ensure the safety of cannabis and hemp products by testing for heavy metal contamination using ICP-MS and state specific reference material mixes

Full workflow applications



MilliporeSigma

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# Thank You

Supelco Products for Analytical Cannabis Testing WFs, Distr trng. - Sept. 10, 2021


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