

# *Analytical Testing of Hemp: Agricultural Certification and Consumer Protection*

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**Understanding the Environment  
for a Sustainable Future**

# Introduction

## Hemp Testing Model

- What is an appropriate model for testing hemp?
  - Agricultural commodities
- Goals of testing:
  - Potency – phytochemical composition and abundance
  - Consumer Protection – screening for contaminants



# Why test hemp?

## Potency

- Is it Hemp?
  - Hemp – delta9-THC  $\leq$  0.3% (mass)
  - Not Hemp - delta9-THC  $>$  0.3% (mass)
- Is it Commercially valuable
  - Cannabinoids
  - Terpenes

## Consumer Protection

- Set standards for manufactured hemp products
- Meant to ensure safety and compliance



# Laboratory Regulations?

## State of CT -- Departments of Agriculture and Consumer Protection

- Controlled Substance Laboratory License
- Have or be working to attain ISO 17025 Accreditation

## Federal Govt – USDA and DEA

- New hemp regulations - some controversy...
- Requiring destruction under law enforcement
- Require analysis at DEA Certified Labs
  - ***No labs in New England (44 nationwide)***



# ISO Laboratories

## What is ISO 17025?

- A system to ensure continuous improvement and self-correction:
  - Competence
  - Impartiality
  - Consistency
- Traceability
- Documentation
- Corrective action
- Customer centric



# ISO Laboratories

*Uncertainty* -- One of the key elements of ISO Accreditation

- The range around a reported result within which the true value can be expected at a certain probability.
- Includes accuracy, repeatability, reproducibility

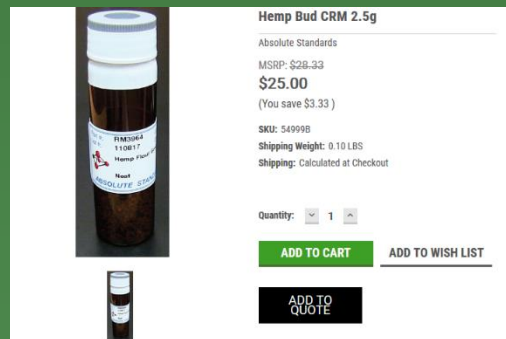
In CT the THC results had a  $\pm 20\%$  uncertainty (up to 0.36%) built in.



# ISO Laboratories

## ISO 17025 Also Ensures Proficiency

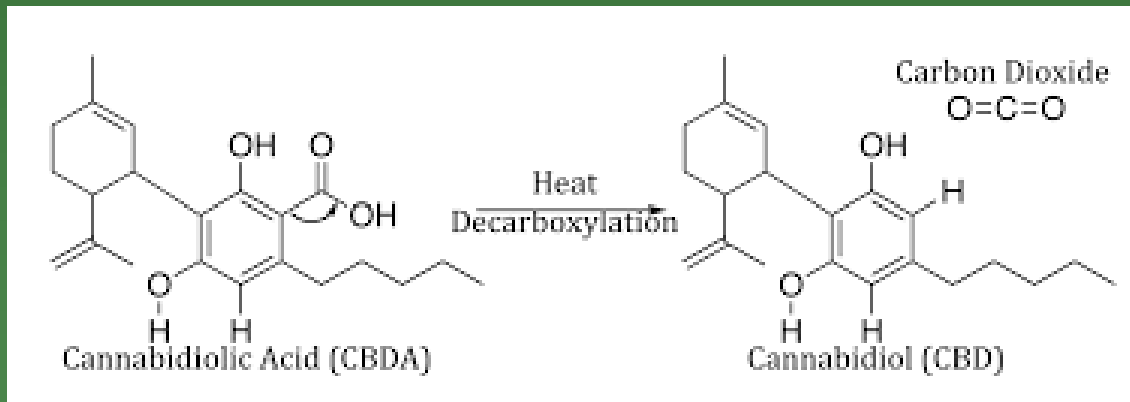
- Proficiency testing - **Unknown**
  - State of KY (THC/CBD only – Sep/Oct)
  - 3<sup>rd</sup> party - Phenova, Emerald Sci.
- Standard reference materials - **Known**
  - Relatively recent
  - Previous were marijuana based



# Potency/CBD Testing

There are 2 Main Types of Compounds Tested:

- THC/CBD compounds and their acidic versions (i.e THC-a/CBD-a)
- The acidic version are the main type in fresh plants
  - Convert (Decarboxylate) due to heat/curing





# Potency/CBD Testing

## Why is This Important?

- The 0.3% THC standard is based upon total Delta-9 THC
- Slightly confounds things is there are 2 main methods for testing
  - HPLC – parent and acidified compounds
  - GC – parent compounds
- How address this issue:
  - Calculation:  $\text{Total THC} = \text{THC} + (0.877)\text{THC-a}$
  - Heat: force decarboxylation via heat



# Potency/CBD Testing

## Preparation Procedures

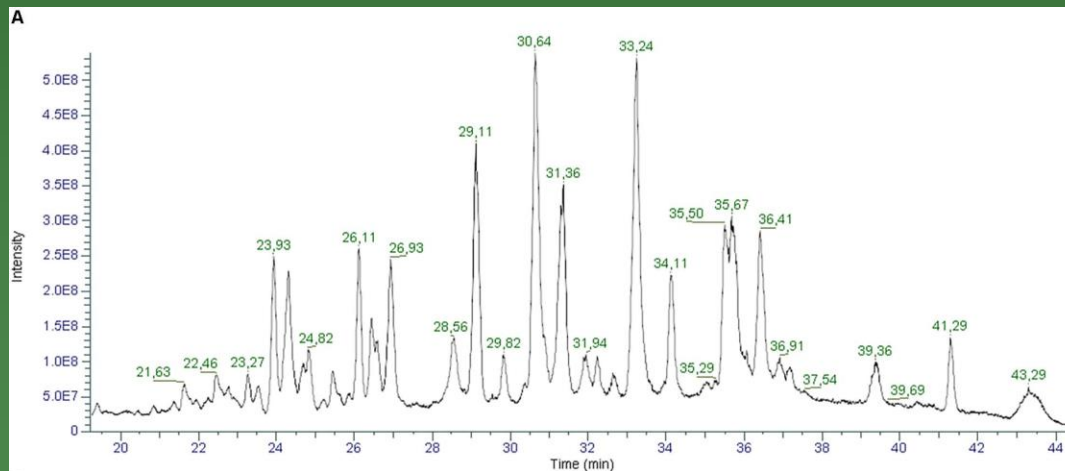
- Dry at 90C for 18-24 hours
- Record weights to ensure consistent dryness
- Separate the stems and seeds via #10 sieve
- Pulverize in a specimen mill until completely uniform in particle size



# Potency/CBD testing

## Analytical Challenges

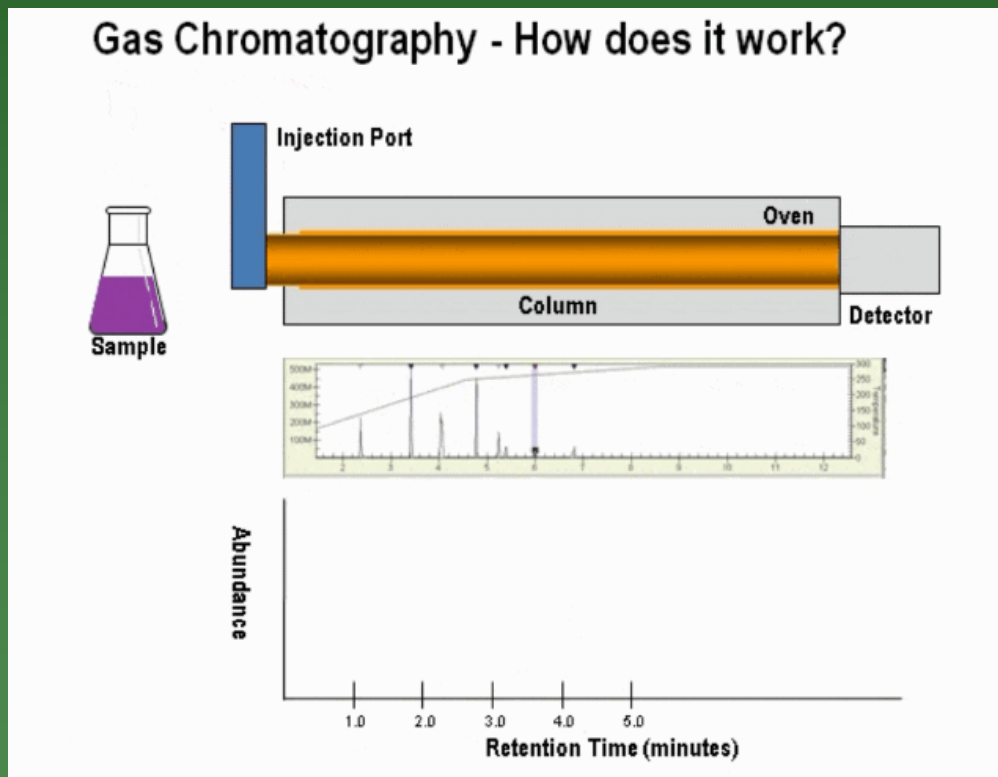
- Very complex – thousands of compounds
  - Many bioactive
- Need to separate the “wheat from the chaff”
- Chromatography very important



# Potency/CBD Testing

## What is Chromatography

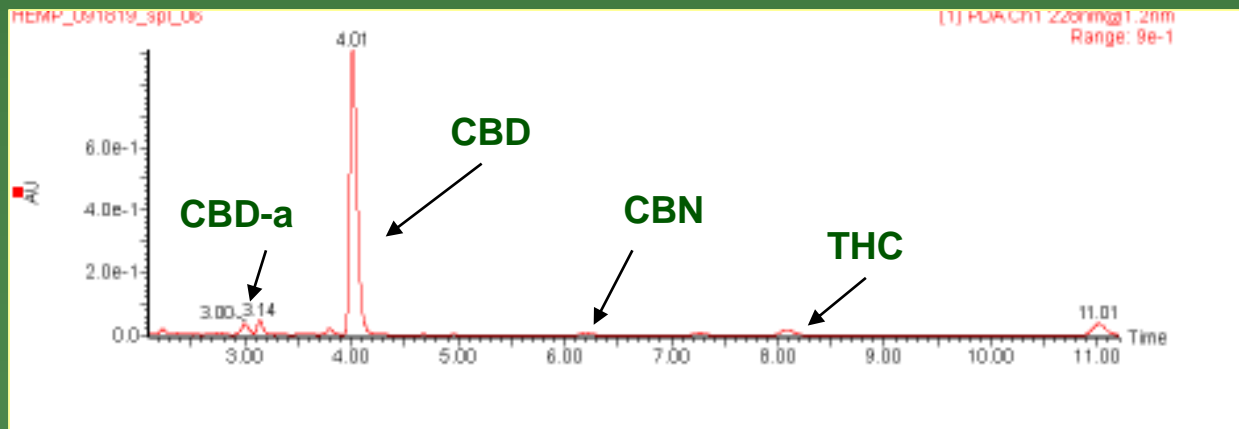
- Separates compounds based on size, charge, how chemical binds to column



# Potency/CBD Testing

## Ultra High Pressure Liquid Chromatography

- Method CESE uses
  - Allows for quantitation of THC/CBDs and acid forms
  - Faster run times
  - MS/MS or UV detection
- Variable concentrations
  - High level of sensitivity
  - 2 dilution levels



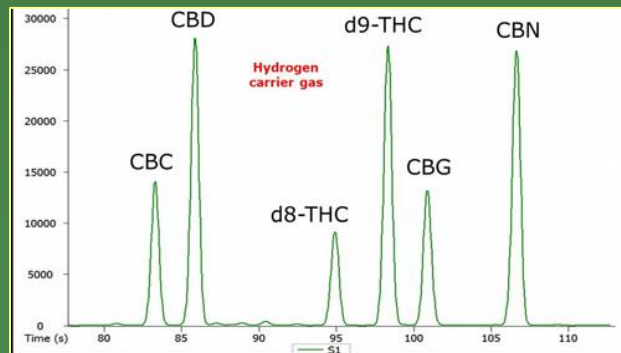
# Potency/CBD Testing

## Gas Chromatograph – Flame Ionization

- Commonly used method
  - Converts to decarboxylated forms
  - Longer run times
  - Less sensitivity

## Gas Chromatograph – Mass Spec.

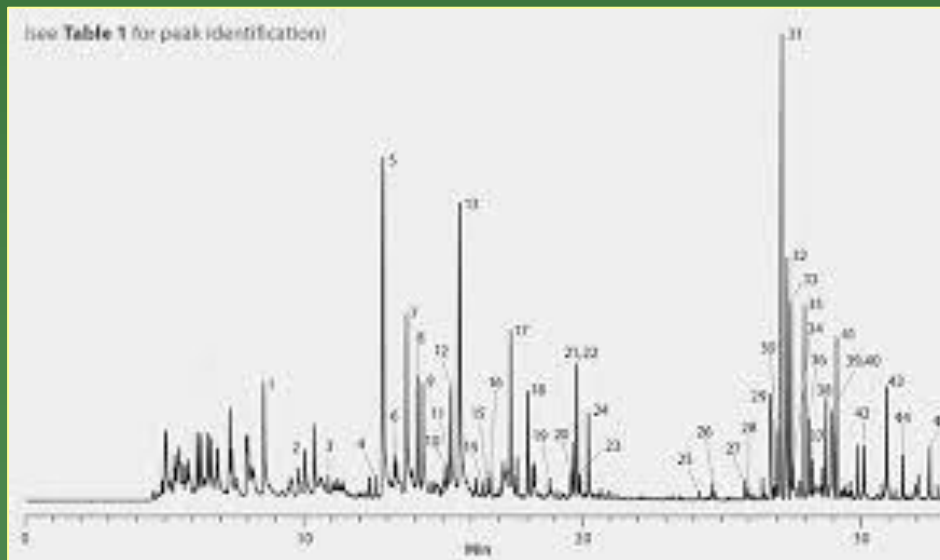
- More sensitive
- Less subject to interference



# Potency Testing

## Terpenes

- Aromatic compounds (volatile – heat loss)
- Over 100 found in *Cannabis spp.*
- Add value to the product – entourage effect
- Analysis by gas chromatography
- We report the 8 most common compounds



# Product Safety Testing

## Consumer Safety – Dept. of Consumer Protection

- Pesticides
  - Analysis for those approved for use in CT
- Metals
  - Cd, As, Hg, Pb
- Microbes
  - Molds and Bacteria
- Mycotoxins
  - Aflatoxin, Ochratoxin





# Product Safety Testing

## Pesticides Testing

- Tremendous variability between states
  - CA 66 pesticides – can be expensive
- In CT – Not clearly defined in Regulations
  - Based upon DoAg Approved List – active ingredients
  - Many are natural products
  - Pyrethrins, Azadirachtin, piperonyl butoxide



# Product Safety Testing

## Mycotoxin testing

- Several analysis methods
  - LC/MS/MS
  - qPCR
- Higher level of sensitivity required
  - 20 ug/Kg (20 ppb)
- Able to combine with pesticide test to be more efficient and cheaper if do both together.



# Product Safety Testing

## Metals Testing

- Utilize very different instrumentation - ICPMS
- Uses plasma (~6,000 K) to break down to ions
- Part per million range
  - 2.7 (Cd) – 8.7 (Pb/Hg)



# Product Safety Testing

## Microbial Testing

- 2 Main Methods used
  - Traditional Agar Plate
  - qPCR
- qPCR faster (several hours vs days)
- Specificity – target microbes only
- More precise and happens in real-time
- Can analyze multiple pathogens simultaneously
- More expensive to run



# Product Safety Testing

## Consumer Product Testing – *Recent Work*

- Working with several customers -- CBD
- Several different matrices
  - Lotions
  - Oils – olive - coconut
- This has unique challenges
  - Each matrix can behave differently – requiring different preparation methods
- Retailers – CBD % validation
- Manufacturers - CoA



# Lessons Learned

## Rapid gear up to meet the DoAg testing needs

- Streamlined preparation methods
- Gain redundancy on THC/CBD
- Increase sample throughput and reporting
- Continue work on our ISO accreditation
- Increase our consumer product methods



**Questions???**

