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 +/- 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)
  - 3rd 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: Total THC = THC + (0.877)THC-a
  • Heat: force decarboxylaion 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
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

Image: Chromatogram indicating CBD, CBN, THC, and CBD-a peaks.
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
  • Alflatoxin, 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 done 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

**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???