

Laboratory analysis of processed foods, raw agricultural commodities, and animal feeds in Connecticut

DEPARTMENT OF ANALYTICAL CHEMISTRY

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History

The Connecticut Agricultural Experiment Station

- Analytical testing lab for Connecticut
- Founded in 1875, moved to present location in 1882
- Fertilizer analysis
- Food adulteration/analysis
- Pesticide analysis began in 1930s
 - Inorganic pesticides, lead and arsenic
 - DDT in 1945



Johnson-Horsfall Building

Analytical Chemistry

Food Emergency Response Network (2005)

- Effort to respond to potential threats to food supplies
- 1 of the 8 original laboratories

FERN assignments:

- Melamine in pet foods and other foods with wheat gluten
- Imported toothpastes with ethylene glycol and diethylene glycol
- PAHs in seafood from Deepwater Horizon spill
- Arsenic speciation in apple juice



ISO 17025 accreditation

Evaluation of quality management system and technical competency to carry out tests and calibrations.

Accreditation achieved in 2016.

Pesticides, aflatoxins, total THC, total CBD, arsenic, crude fiber, and crude protein are analyzed under scope.



Sample types

Fresh agricultural commodities

- Fruits
- Vegetables

Animal Feeds



Sample login



Connecticut Department of Consumer Protection,
Food & Standards Division
450 Columbus Boulevard Ste 901 Hartford, CT 06103
www.ct.gov/dcp

Connecticut 484

Insp Date: 11/9/2021 Business ID: 00003863
Business: MARKET 32 BY PRICE CHOPPER #248
140 Main St

Inspection: CP000981
License No.: BAK15992
Phone: 203-376-7182
Inspector: 965901 Keara Hickey
Reason: Sample Collection
Results: Complete

OXFORD, CT 06478

Reference:

Sample Collection Identification			
Sample No.	KH-180	Complaint No.	2021-202
Type of Product	Asparagus		
Sample Collection Purpose			
Reason Sample Collected	Pesticide Residues Surveillance		
Other Reason Sample Collected:			
Analysis Requested:	Pesticides		
Sample Collection			
Sample Size	Net Weight of	N/A	Quantity
			2 bundles
	2 bundles taken from total of 12 in one-11lb box. 4 boxes total		
Total Lot			
Method of Collection	Non-Gloved Manual Collection		
Special Techniques	Yes <input type="radio"/>	No <input checked="" type="radio"/>	
Location & Temperature	Location Sample	Walk-in Cooler	Sample
	Temperature at	5.0 °C	Open <input checked="" type="radio"/> Unopened <input type="radio"/>



Sample LogBook

Station Number: 21. 5950

Sample Number
KH-180

Save and Exit

1) Inspector Info 2) Sample ID 3) Details 4) Analysis Request 5) Internal CoC

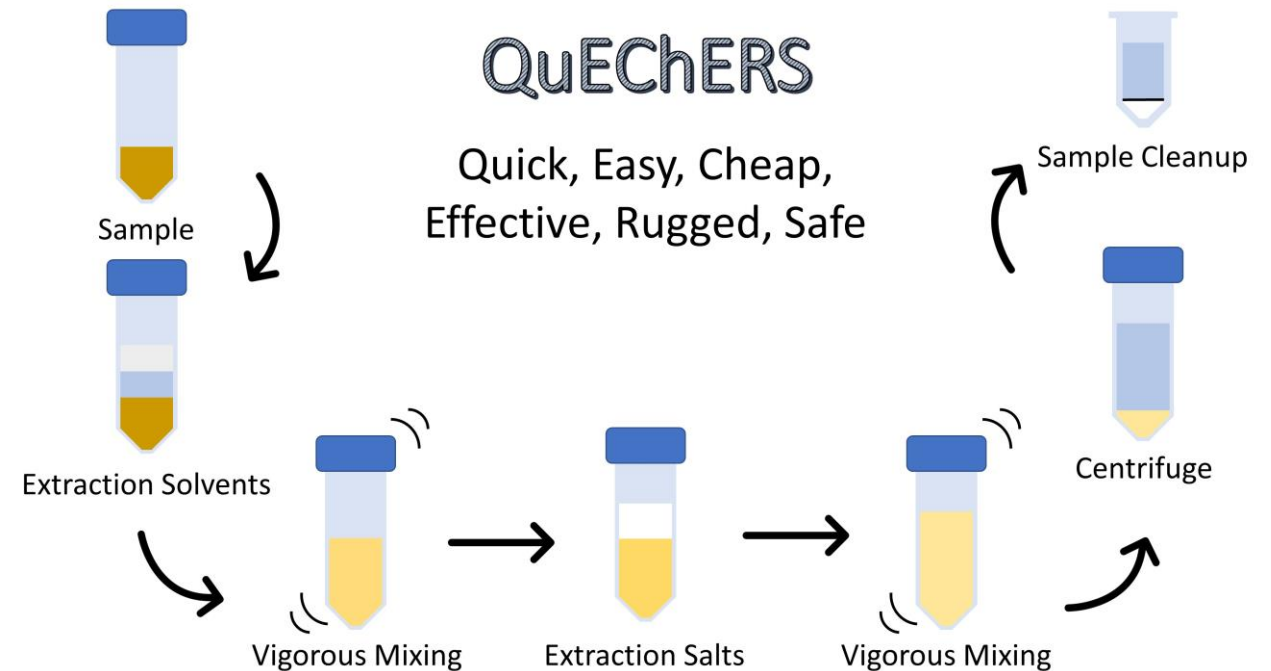
Date Collected:	11/9/2021	Collection Report	Keara Hickey Department of Consumer Protection Food Division
Sample Number:	KH-180	New Sample Click here	Save and Exit
Sample Type:	Raw Agricultural Commodities		
Storage Preference:	Refrigerated		
<input type="radio"/> ISO 17025 compliance samples <input type="checkbox"/> PT sample/ check sample ? <input type="checkbox"/> Research Sample ?	Sub-samples: 1		
Sample Description (On label or any deviation from collection report:)	Fresh asparagus Hydrocooled PLU 4080		
Collection and Inspector Reports	file:///R:\Analytical\Accredited%20files\Samples\Collection%20Reports\KH-180%20to%20KH-189%20Reports.pdf		
Note (will be on the report) ----->	Fresh		
Organic ? Processed? Fresh? High Priority?			
Laboratory Note:			

Pesticides

Fresh samples are ground and homogenized

Extracted using acetonitrile

Clean up with $MgSO_4$ and $NaOAc$



Pesticides

Samples analyzed using a Liquid Chromatography High Resolution Mass Spectroscopy and Gas Chromatography tandem mass spectroscopy.



Scope of accreditation covers over 350 pesticide residues.

Pesticide tolerance levels

Who decides?

- EPA decides how much pesticide can remain on in foods—tolerances.
- Correctly applied pesticides are unlikely to leave residues over tolerance.
- Levels below tolerance are unlikely to cause harm.



Pesticide tolerance levels

How are levels decided?

- “Reasonable certainty of no harm.”
- Registrants must submit studies for EPA review.
 - Possible harmful effects
 - Amount likely to remain on food
 - Other sources of exposure
- EPA performs risk assessment
 - Food consumption
 - Pesticide application patterns



Code of Federal Regulations

A point in time eCFR system



No tolerance violations

Accidental or deliberate application to crops on which they are not permitted.

31 out of 945 samples tested for pesticides (since 2015)

22 unique residues

4 samples contained multiple NTVs

Violation rate is approximately 3-4%

Market basket survey continues to demonstrate the safety of fresh produce in CT.

Pesticide	Pesticide
Acephate	Diphenylamine
Bifenthrin	Endosulfan
Carbendazim	Metalaxyl
Carbofuran	Methamidophos
Chlorpropham	Oxyflurofen
Chlorpyrifos	Phosmet
Cyfluthrin	Propargite
Cyhalothrin, lambda	Tebuconazole
Dicloran	Thiabendazole
Difenoconazole	Triadimefon
Dimethoate	Triadimenol

Carbofuran in asparagus

Imported from Peru

Carbofuran found at 0.03 mg/kg

FDA placed foreign processor on Import Alert 99-05

- *Detention without physical examination (DWPE)*

AGROEXPORTACIONES NATHANAEL S.A.C

Date Published : 03/11/2022

Cal. Victor Raul Nro. S/N C.P.M Macabi B , Ascope - Pajjan, La Libertad PERU

24 T - - 02 Asparagus (Leaf & Stem Vegetable)

Date Published: 03/11/2022

Desc: Asparagus

Problems: CARBOFURAN;



No tolerance violations (2022)

Green seedless grapes

- Imported from Peru
- Diphenylamine, 0.19 mg/kg



Fresh ginger

- Imported from Brazil
- Chlorpropham, 0.06 mg/kg



Fresh ginger

- Imported from Honduras
- Phosmet, 0.03 mg/kg

No tolerance violations (2022)

Italian Parsley

- Grown in California
- Chlorpropham, 0.151 mg/kg

Dragon fruit

- Imported from Ecuador
- Thiabendazole, 0.04 mg/kg

Yellow yam

- Imported from Jamaica
- Dicloran, 14.80 mg/kg



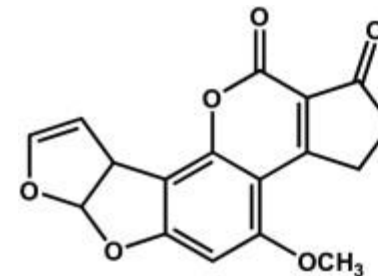
Aflatoxins

Carcinogenic toxins produced by fungus *Aspergillus flavus*

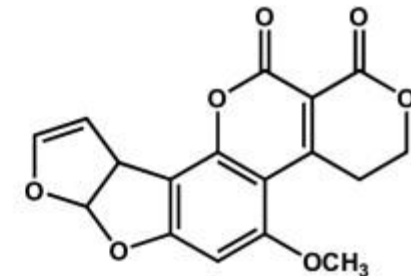
- Can grow on cereal grains and legumes.

FDA has an action level based on B1 + B2 + G1 + G2

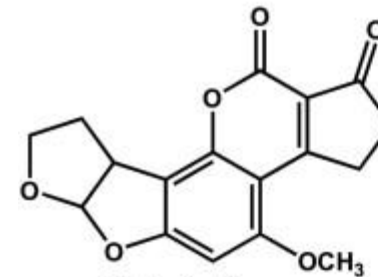
- 100-300 $\mu\text{g}/\text{kg}$ for beef cattle, poultry, and swine feed.
- 20 $\mu\text{g}/\text{kg}$ for dairy/immature animals and pets.
- CAES has a 1 $\mu\text{g}/\text{kg}$ limit of reporting.



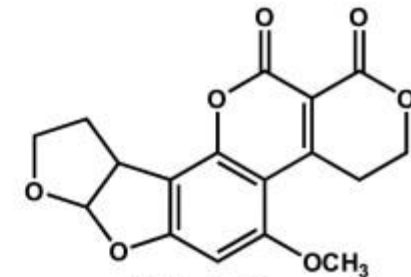
Aflatoxin B₁



Aflatoxin G₁



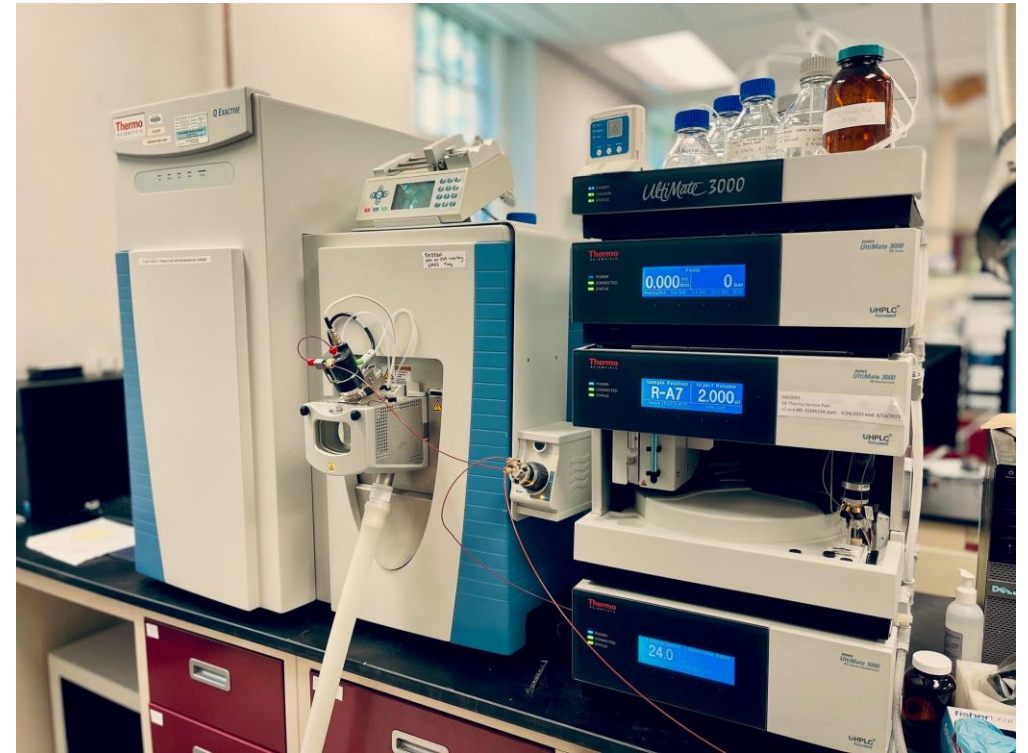
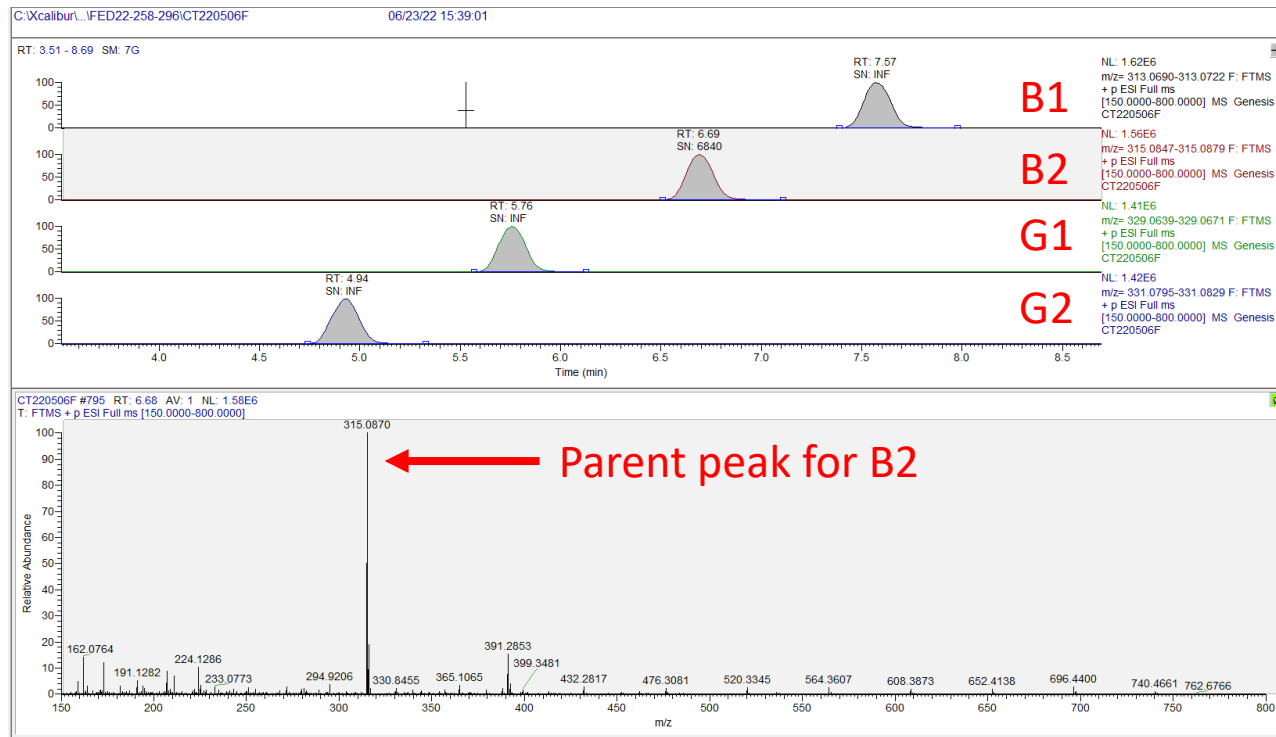
Aflatoxin B₂



Aflatoxin G₂

Aflatoxins

Samples extracted using 80% acetonitrile in water, followed by SPE with immunoaffinity cartridges specific for aflatoxins.



Aflatoxins

61 animal feed products tested in 2020

- No aflatoxins reported

148 animal feed products tested in 2021

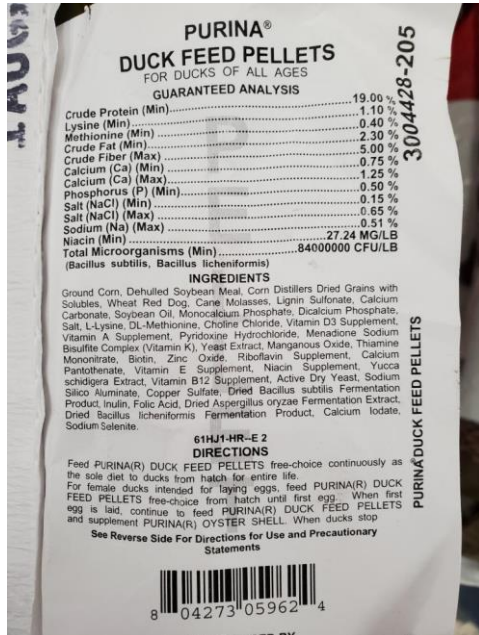
- 2 products reported with $< 2 \mu\text{g}/\text{kg}$.



Label guarantees

Ensure manufacturer label accuracy

- Crude protein
- Crude fat
- Crude fiber



Label guarantees

In 2020, 44 samples tested:

- 3 were unsatisfactory for at least one macronutrient
 - 1 crude protein
 - 2 crude fat

In 2021, 51 samples tested:

- 4 were unsatisfactory for at least one macronutrient
 - 1 crude fiber
 - 1 crude protein
 - 2 crude fat

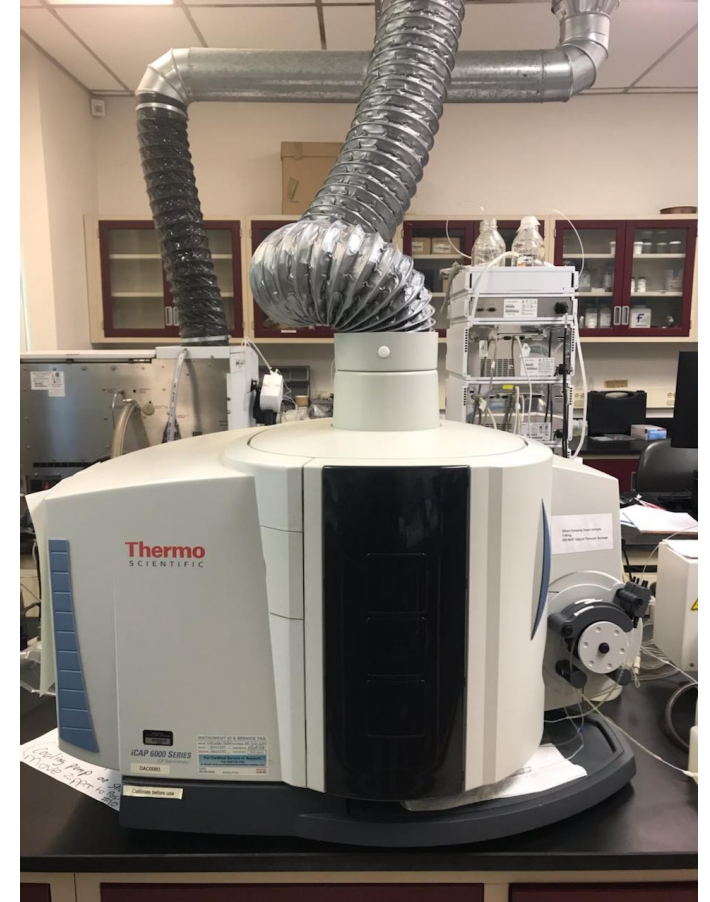


Metals

Nutrient content in food

Total arsenic in juice and foods

Heavy metals in food products



Cannabis sativa

Agricultural Improvement Act of 2018

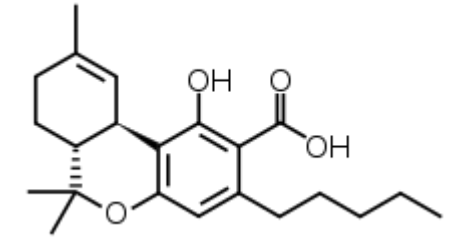
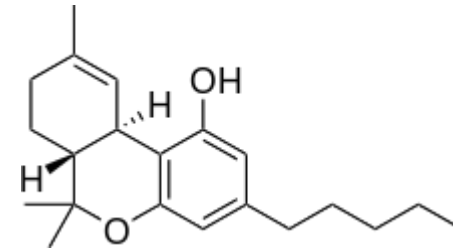
- Hemp must contain $< 0.3\%$ THC.

Representative samples must be shown to meet Farm Bill requirements prior to harvest.

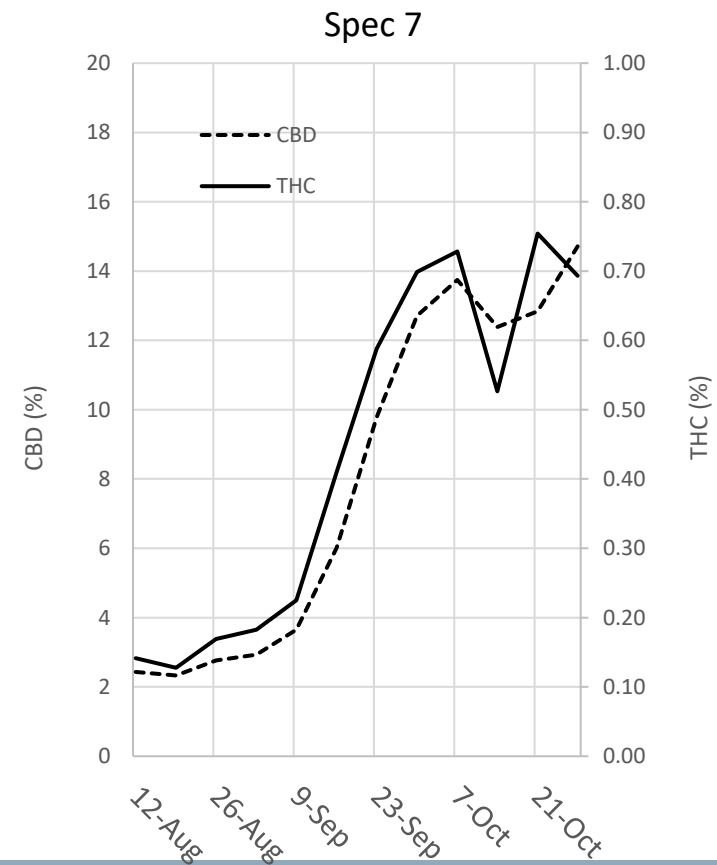
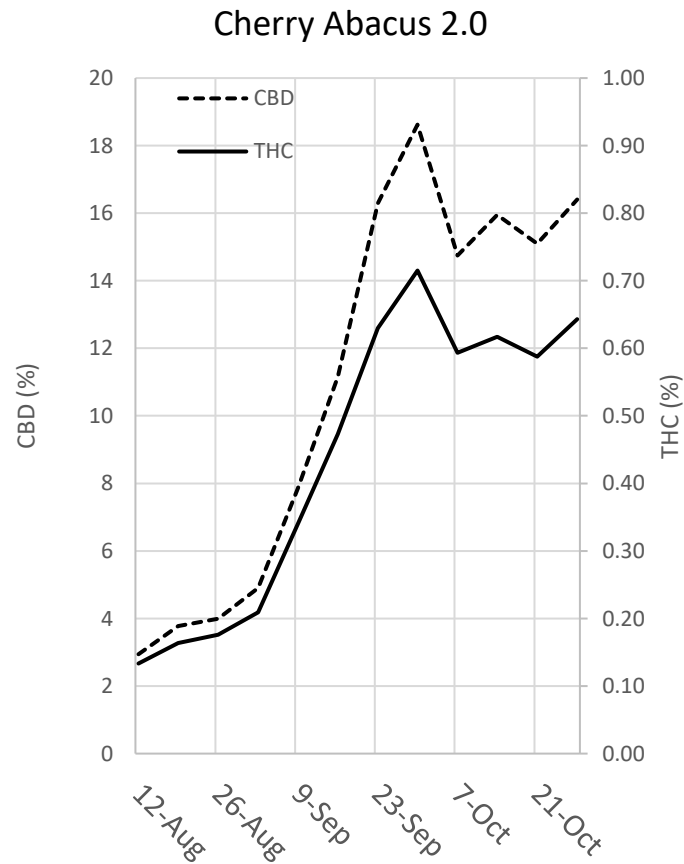
Starting with 2022 season DoAg inspectors will collect preharvest samples for THC analysis



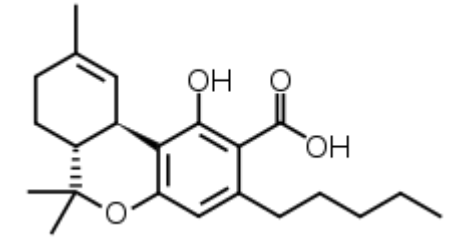
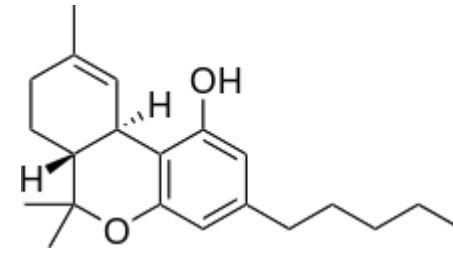
Cannabis sativa



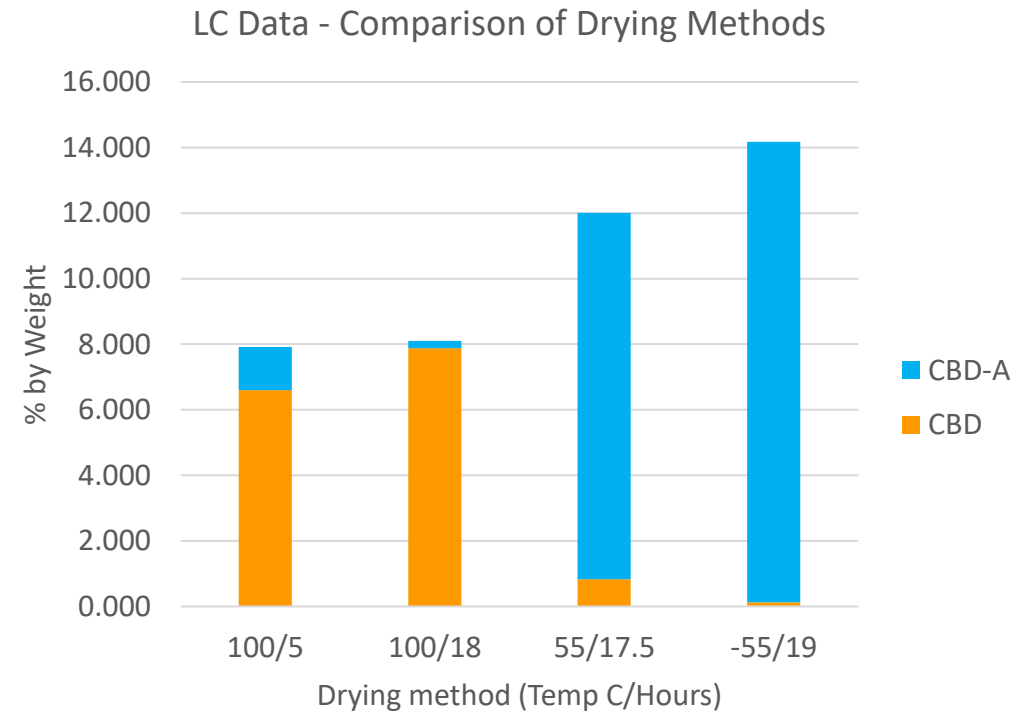
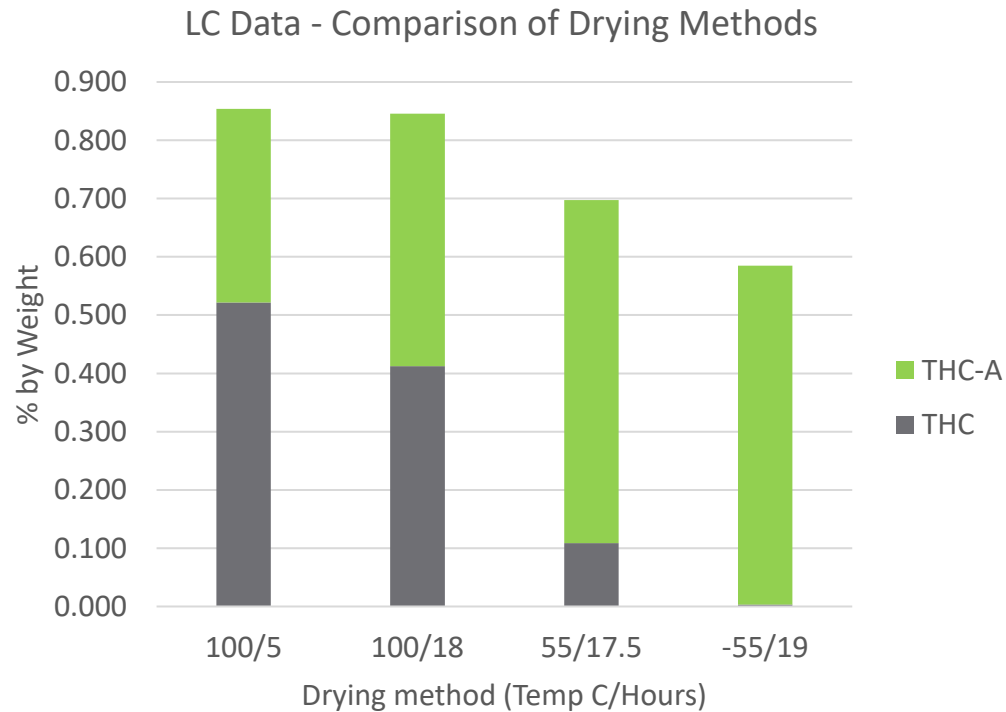
Weekly monitoring of THC and CBD showed that in many varieties THC and CBD rise in tandem



Cannabis sativa



Effects of drying temperature on delta 9 THC and CBD



Higher temperature, and longer time, decarboxylate THC-A and CBD-A

Acknowledgments

This work represents the efforts of DAC:

- Terri Arsenault
- Kitty Prapayotin-Riveros
- Mike Ammirata
- Meghan Cahill
- John Ranciato
- Craig Musante
- Brian Eitzer
- Walter Krol
- Chris Dimkpa
- Jason White



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