Avocado

– recipe or working method?

WLODEK

Wlodzimierz S. BOREJSZA-WYSOCKI Ph.D.
IR-4 Southern Regional Laboratory Research Director

Food & Environmental Toxicology Laboratory
Institute of Food and Agricultural Sciences
University of Florida, Gainesville, FL 32611

Orlando, FL, 2/28-3/01/2017

Perfect Guacamole ?

Perfect IR-4 sample

Projects

6-Benzyladenine
(2012), PR#10922

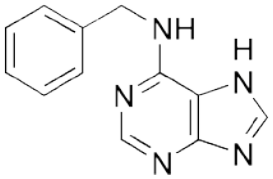
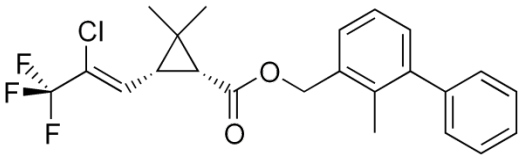
Bifenthrin
(2013), PR#10578

AVOCADO

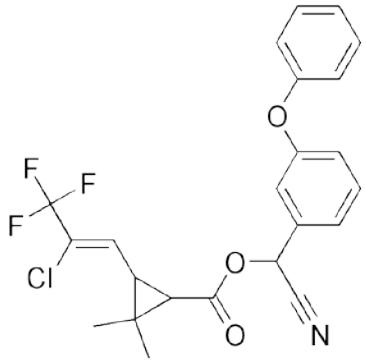
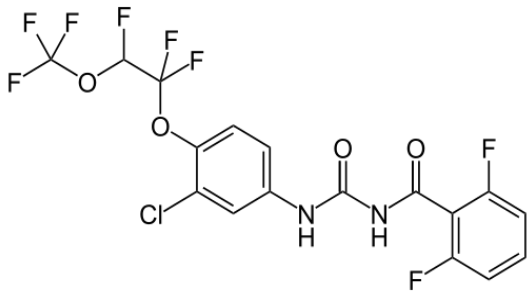
λ-Cyhalothrin
(2010), PR#10540

Novaluron
(2009), PR#09246

Reference Method

CHEMICAL	STRUCTURE	COMMODITY	EXTRACTION SOLVENT (Relative polarity)
6-BENZYLADENINE **(Plant Growth Regulator)** (insoluble in water)		**Apple and Pear**	**0.1 N HCl in Methanol (0.762)**
BIFENTHRIN **(Insecticide)** (poorly soluble in water 0.1 mg/L)		**Potato**	**Acetone (0.355)**

Reference Method

CHEMICAL	STRUCTURE	COMMODITY	EXTRACTION SOLVENT (Relative polarity)
λ-CYHALOTHRIN **(insecticide)** (insoluble in water 0.005 mg/L)		**Fruits, vegetables and grains**	**Acetone:Hexane** **50:50** **(0.182)**
NOVALURON **(insecticide)** (insoluble in water 0.003 mg/L)		**Pome Fruits**	**Methanol:Water** **70:30** **(0.881)**

Commodities

Commodity	Compounds (%)			
	Water	Fat	Carbohydrates	Protein
Avocado	**73**	**15**	8.5	2.0
Apple & Pear	86 84	0.2 0.1	13.8 15.2	0.3 0.4
Potato	79	0.1	17.5	2.0
Cauliflower Broccoli Cabbage	92 89 90	0.3 0.4 0.1	5.0 6.6 5.8	1.9 2.8 1.3
Bean Pea	79 79	0.5 0.4	10.5 14.5	9.6 5.4

Modifications
(6-Benzyladenine on Avocado)

Reference Method	Working Method
*Acidic Partition and Basic Partition	
3 x partitioned vigorously shaken	4 x partitioned gently shaken (reduced emulsion formation)
*Derivatization	
Yes	No
*Post-Derivatization Cleanup	
Yes	No
*Instrument	
GC/MS	LC/MS/MS

Modifications
(Bifenthrin on Avocado)

Reference Method	Working Method
* Extraction	
2.5 g + 2 x 15 mL acetone	2.5 g + 2 x 15 mL acetone
* Evaporate under a stream of nitrogen (~35oC)	
~ 2 mL residual water	~2 mL 2mL residual water + oil
* Partition with hexane	
Yes	Yes
* Cleanup	
1 g Silica SPE cartridge	2 g Silica SPE cartridge
*Elute Bifenthrin with	
12 mL of 10 % ethyl acetate/hexane	4 mL of 4% ethyl acetate/hexane (discard), 7 mL of 4% ethyl acetate/hexane

* Evaporate to ~ 0.25 mL remains under nitrogen, dissolve in hexane and analyze by

GC/MSD	GC/ECD

Modifications
(Novaluron on Avocado)

Reference Method	Working Method
* Extraction	
2 x 100 mL Methanol : water (70:30)	2 x 100 mL Methanol : water (70:30)
	1 x 50 mL Methanol
* Evaporate on rotary evaporator at ~40oC	
*Liquid/Liquid cleanup	
2 x with hexane	2 x with ethyl acetate
* Evaporate on rotary evaporator at ~40oC	
to a low volume (hexane)	to “dryness” and add ethyl acetate
* SPE cleanup on NH_2 cartridge	
Loaded and discarded	loaded and collected
Elute with diethyl ether : ethyl acetate (50:50) and acetone : hexane (50:50)	elute with ethyl acetate

*Evaporate under a stream of nitrogen and reconstitute in the ethyl acetate

*Analyze the Novaluron residue by GC/ECD

Modifications
(λ-Cyhalothrin on Avocado)

Reference Method

* Extraction with hexane : acetone (50:50)

* Evaporate on rotary evaporator

complete dryness

↓

add 4 mL acetonitrile, sonicate

add 6 mL water , sonicate

(dilute with 60/40, water/acetonitrile)

↓

* Instrument

LC/MS/MS

Working Method

* Extraction with hexane : acetone (50:50)

* Evaporate on rotary evaporator

"near dryness" (oil/water)

↓

* Solvent exchange -add hexane, sonicate

* Centrifuge

Separate hexane (add sodium sulfate)

↓

* Liquid-Liquid Extraction Cleanup

add acetonitrile saturated with hexane

↓

separate acetonitrile, evaporate to dryness

and residue dissolve in hexane

↓

* SPE Cleanup

Modifications
(λ-Cyhalothrin on Avocado)

Reference Method

Working Method

↓

*** SPE Cleanup**

2 g Cyano Cartridge

↓

Elute with 1.5% acetone in hexane

↓

Evaporate to dryness

↓

Dissolve residue in 10 mL acetonitrile (sonicate), add 15 mL water and mix well (60/40 water/acetonitrile)

↓

*** Instrument**

LC/MS/MS

How much does the working method reflect the reference method ?


Thank you