

Agilent GC/MS Solutions for Cannabis and Hemp Testing

Simon Jones
GCMS Application Scientist



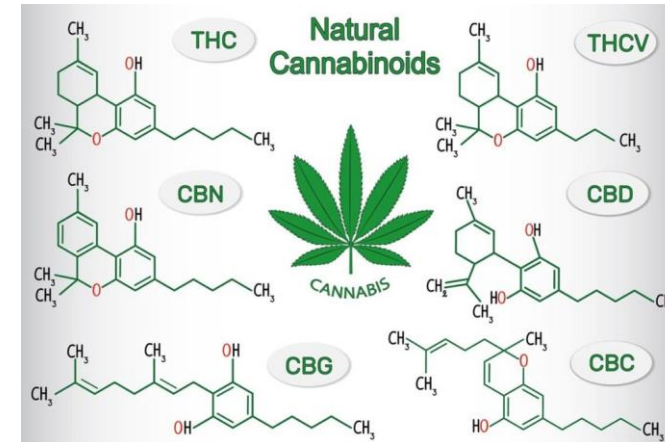
Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

What is Cannabis?

Cannabis Sativa Plant



- Cannabinoids (THC, CBD,)



Click to add text

- Aromatics (Terpene, Sesquiterpenes and other Volatiles)

TERPENES IN CANNABIS

NAME	AROMA	MAINEFFECTS	FOUND IN
MYRCENE	Musky, Herbal	analgesic antioxidant relaxing	wild thyme hops lemon grass
LIMONENE	Lemon-like	anti-inflammatory antioxidant relaxing	citrus fruits rosemary coniferous trees
CARYOPHYLLENE	Spicy, Peppery	analgesic antioxidant neuroprotective	black pepper cloves rosemary/hops
TERPINOLENE	Piney, Herbal	anti-inflammatory antioxidant sedative	allspice parsnip apples
PINENE	Piney	anti-inflammatory antimicrobial antiseptic	pine coniferous tree resin citrus fruits
HUMULENE	Hoppy	anti-inflammatory antibacterial appetite suppressor	hops coriander basil
LINALOOL	Floral	analgesic antimicrobial sedative	lavender mint cinnamon

All images, artwork, text and graphics are the property of Vaping360. All rights reserved. VAPING360

What is Cannabis?

Plant *Cannabis Sativa*

✓ Diverse and Varied Use

- Recreational : beer, oil, cakes, joint



- Medicinal : Pain Killer / anti-inflammatory



- Others : textile, isolation, feed...



System Platform (Intuvo)

Common and Optional Features

Common to all 3 Intuvo methods

- MultiMode Inlet (MMI), Guard Chip, Mid-Column Backflush
- Matrix matched (cannabis and oils)
- Use of Internal standards

Common to RSA and Terpenes

- 2 DB-Select 624 Ultra Inert columns
- Low pressure drop, glass wool split liner
- MS operated in EI SIM mode
- 5977B MSD with an Extractor EI Ion Source and a 9 mm Extractor Lens

Optional for RSA & Terpenes

- XLSI Transfer Liner Interface (G3969A)
- 7650A Automatic Liquid Sampler (G4567A)
- S/SL Inlet and Guard Chip (G4587-60565)



Lots of Uses, But What is Tested..... By GCMS?

Pesticides

GC 3Q MS

Residual Solvents

GC single Quad MSD

Redundant System for Terpenes

Terpenes

GC Single Quad MSD

Redundant System for RSA



Background information

This eMethod is designed for the analysis of 17 GC-MS/MS amenable pesticides regulated by Health Canada. The eMethod defines single stream (LC-MS/MS and GC-MS/MS) sample preparation and includes a list of expertly selected consumables and supplies. It also provides an optimized analytical method for the separation and reliable detection of the targeted pesticides under 16 minutes. The same method used in the application note provided excellent calibration, recoveries, and LOQ data to demonstrate the reliable measurements of all GC-MS/MS amenable pesticides.

Instrument configuration

Agilent 8890 GC with the 7010B Mass Spectrometer and 7693 Automatic Liquid sampler. (ALS)

The GC includes MMI inlet and mid-column backflush. The 7010B MS was configured in EI mode. MassHunter 10.0 was used both for acquisition and data analysis.



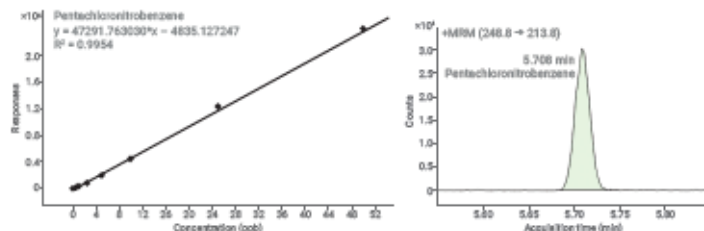
GC/MS Pesticide Residue Analysis: Canadian GC/MS/MS eMethod

Application note



A Sensitive and Robust Workflow to Measure Residual Pesticides and Mycotoxins from the Canadian Target List in Dry Marijuana Flower

Calibration and Detection of Pentachloronitrobenzene (Quintozene)



Includes all GC/MS amenable pesticides monitored in Canada (Does not include Captan!)

Designed for accuracy and robustness

16 minutes run time

For more information visit <http://www.agilent.com/chem/cannabis-testing-emethods>

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

DE 5367476852

This information is subject to change without notice.

© Agilent Technologies, Inc. 2020
Published in the USA, June 1, 2020
5994-2107EN

GC/MS Pesticide Residue Analysis

The Fast Five – Limited US eMethods

Background information

This eMethod is designed for the analysis of 5 GC/MS/MS amenable pesticides regulated by several US states. It defines single stream (LC-MS/MS and GC-MS/MS) sample preparation and includes a list of expertly selected consumables and supplies. It also provides an optimized analytical method for the separation and detection of pentachloronitrobenzene, parathion-methyl, captan, chlordane and chlorfenapyr under 15 minutes. The reliable operation of the end-to-end workflow is demonstrated by excellent calibration results ranging from 2 to 8,000 ppb in matrix, precision, accuracy and LOQ data as described in the application note.

Instrument configuration

Agilent Intuvo 9000 GC with the 7010B Mass Spectrometer and 7693 Automatic Liquid sampler (ALS)

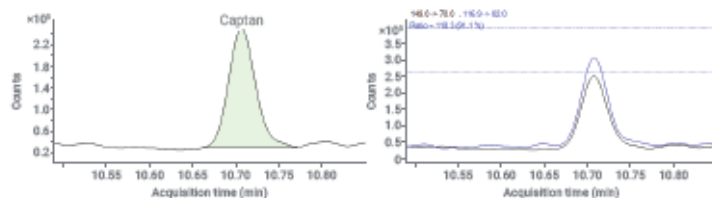
The GC includes MMI inlet and mid-column backflush. The 7010B MS was configured in EI mode. MassHunter 10.0 was used both for acquisition and data analysis.



Application note

 Analysis of Challenging Pesticides Regulated in the Cannabis and Hemp Industry with the Agilent Intuvo 9000/7010 GC/MS/MS System: The Fast-5

Captan Detection in Matrix at 1 ppb Concentration (in vial)



Includes only 5 GC/MS amenable pesticides, but that is adequate in some US states, like California

Small footprint, but robust results

Faster analysis due to shorter analyte list (15 min)

For more information visit <http://www.agilent.com/chem/cannabis-testing-emethods>

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

DE 5359143519

This information is subject to change without notice.

© Agilent Technologies, Inc. 2020
Published in the USA, June 1, 2020
5994-2106EN

Residual Solvent Testing

It's not USP 467!

Cannabis is different – It's not one Active Pharmaceutical Ingredient

Regulations are state dependent

Expanded list of compounds – Propane, Butane, Ethylene Oxide...



Expanded action limits

Method USP 467: 2 ppm – 3,880 ppm

Cannabis/Hemp: 1 ppm – 5,000 ppm



Residual Solvent Testing: GC/MS

California (Oct. 2019)
has the strictest
requirements

eMethod will meet CA
requirements for
concentrates, oils

eMethod: 21 VOCs;
Intuvo/5977 with
headspace sampling

Category I Residual Solvent or Processing Chemical	CAS No.	Cannabis Product or Pre-Roll Action or Level (µg/g)	
1,2-Dichloroethane	107-06-2	1.0	
Benzene	71-43-2	1.0	
Chloroform	67-66-3	1.0	
Ethylene oxide	75-21-8	1.0	
Methylene chloride	75-09-2	1.0	
Trichloroethylene	79-01-6	1.0	

**Category I:
1ppm!**

Category II Residual Solvent or Processing Chemical	CAS No.	Cannabis Product or Pre-Roll Action or Level (µg/g)	
Acetone	67-64-1	5000	
Acetonitrile	75-05-8	410	
Butane	106-97-8	5000	
Ethanol	64-17-5	5000	
Ethyl acetate	141-78-6	5000	
Ethyl ether	60-29-7	5000	
Heptane	142-82-5	5000	
Hexane	110-54-3	290	
Isopropyl alcohol	67-63-0	5000	
Methanol	67-56-1	3000	
Pentane	109-66-0	5000	
Propane	74-98-6	5000	
Toluene	108-88-3	890	
Total xylenes (ortho-, meta-, para-)	1330-20-7	2170	

**Category II:
290 ppm
and higher!**

Lowest required level in
Canada: **5000 ppm**

Background information

This eMethod is designed for the analysis of 21 residual solvents in cannabinoid products as listed by the California Bureau of Cannabis Control. The eMethod defines sample preparation and includes a list of expertly selected consumables and supplies. It also provides ready-to-run, optimized analytical methods for the separation and reliable detection of the targeted analytes under 13 minutes. The same method was used in the application note below and provided excellent quantitation from 0.15 to 20ppm for Category I and 20-6000ppm for category II analytes. This exceptional range was achievable by using skillfully selected sample preparation protocols and novel static headspace and GC/MS conditions. Detailed calibration results, intra- and inter-day accuracy and precision along with LOD and LOQ information also presented in the application note.

Instrument configuration

Agilent 7697A Headspace Sampler, the Intuvo 9000GC and 5977B MS.



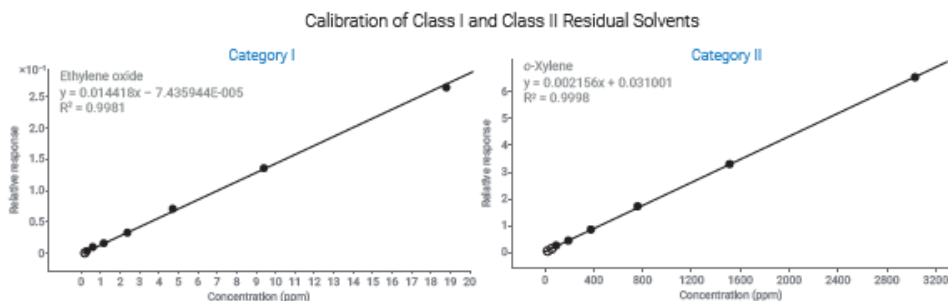
The GC was equipped with a MMI inlet and mid-column backflush.

The 5977B MSD was equipped with an Extractor ion source operated in EI SIM mode. The MassHunter 10.0 SW was used for acquisition and the 10.1 for data analysis.

Detailed application note



Novel Residual Solvents Analysis of Cannabinoid Products with the Agilent Headspace-GC/MS system



For more information visit <http://www.agilent.com/chem/cannabis-testing-emethods>

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

DE.8156092593

This information is subject to change without notice.

© Agilent Technologies, Inc. 2020
Published in the USA, June 1, 2020
5994-2126EN



Reliable detection of analytes with both high and low concentration allowance

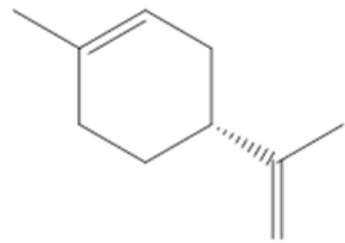
Efficient sample preparation for reliable detection of analytes in a wide Bp range

Prudent system configuration to assure robustness and to avoid contamination from cannabinoids

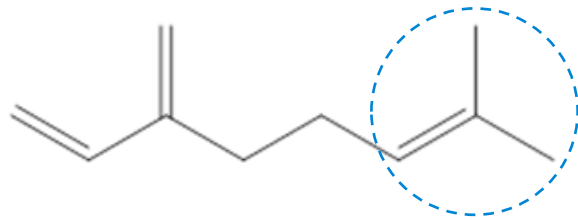
Small footprint

Terpenes – Significance not Fully Understood Yet

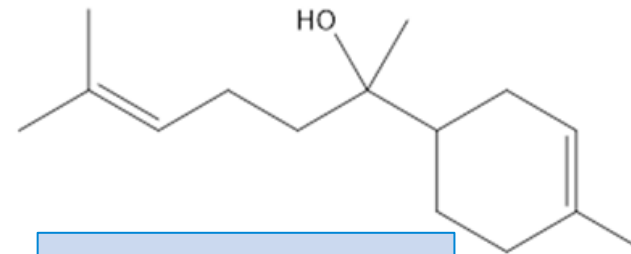
Terpenes are volatile unsaturated hydrocarbons that give cannabis its aromatic diversity. Although these compounds are not psychoactive, they do define the sensory character of the plant. The unique terpene composition gives the signature taste, smell, and experience of a particular strain so regularly reproducing the same terpene profile is key to product consistency.



Limonene
MW: 136



Myrcene
MW: 136



alpha-Bisabolol
MW: 284

Monoterpenes

Sesquiterpene



No regulated target analyte list

If a label includes a terpene as an ingredient, testing is mandatory to verify the analyte and its quantity.

eMethod: 40 terpenes;
Intuvo/5977 with liquid injection

Terpene Detection by Liquid Injection Using the Intuvo/ALS/5977 System

eMethod G5282AA#010

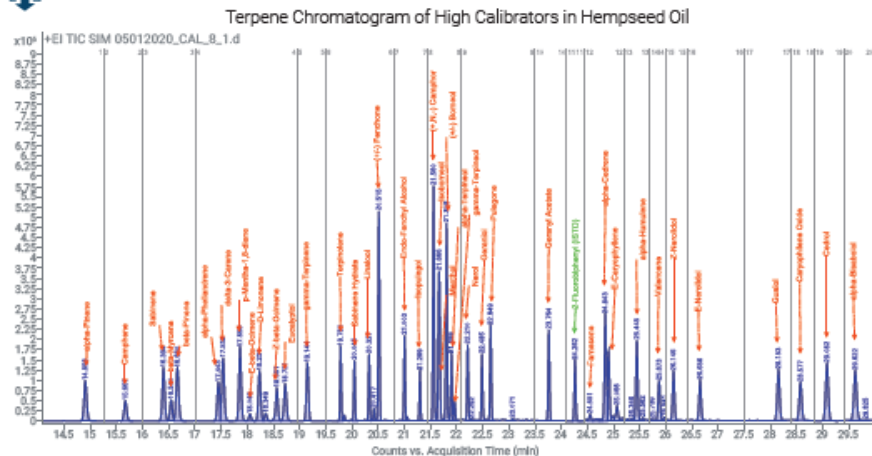
Background information

This eMethod is designed for the analysis of 40 common mono- and sesquiterpenes found in cannabis. The eMethod defines sample preparation and includes a list of expertly selected consumables and supplies. It also provides ready-to-run, optimized analytical methods for the separation and reliable detection of the targeted analytes under 30 minutes, using liquid injection. This technique assures that sesquiterpenoids such as α -bisabolol are accurately detected and not lost as it often happens with headspace sampling. The application note below details sample preparation, matrix matched calibrant preparation with internal standard, regression analysis (~4- 500ug/ml), provides intra- and inter-day precision and accuracy data, LODs and LOQs.

Detailed application note



Terpenes Analysis in Cannabinoid Products by Liquid Injection Using the Agilent Intuvo 9000/5977B GC/MS System



For more information visit <http://www.agilent.com/chem/cannabis-testing-emethods>

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

DE.8193402778

This information is subject to change without notice.

© Agilent Technologies, Inc. 2020
Published in the USA, June 1, 2020
5994-2128EN



Trusted Answers

Terpenes Analysis

Agilent
CrossLab

From Insight to Outcome



Instrument configuration

Agilent Intuvo 9000 GC and the 7650A 50 position Automatic Liquid Sampler connected to a 5977B MSD.

The GC was equipped with a MultiMode Inlet (MMI) and backflush. The 5977B had an InertPlus source and operated in SIM mode. Data were collected with MassHunter B.10 GC/MS Acquisition software, and data analyses were performed with MassHunter Quantitative Software B.10.1.

Sample preparation/introduction to Accommodate reliable detection of sesquiterpenes

Wide calibration range to accommodate naturally occurring terpene concentrations

Prudent system configuration to assure robustness and to avoid contamination from cannabinoids

Small footprint

DE54230972

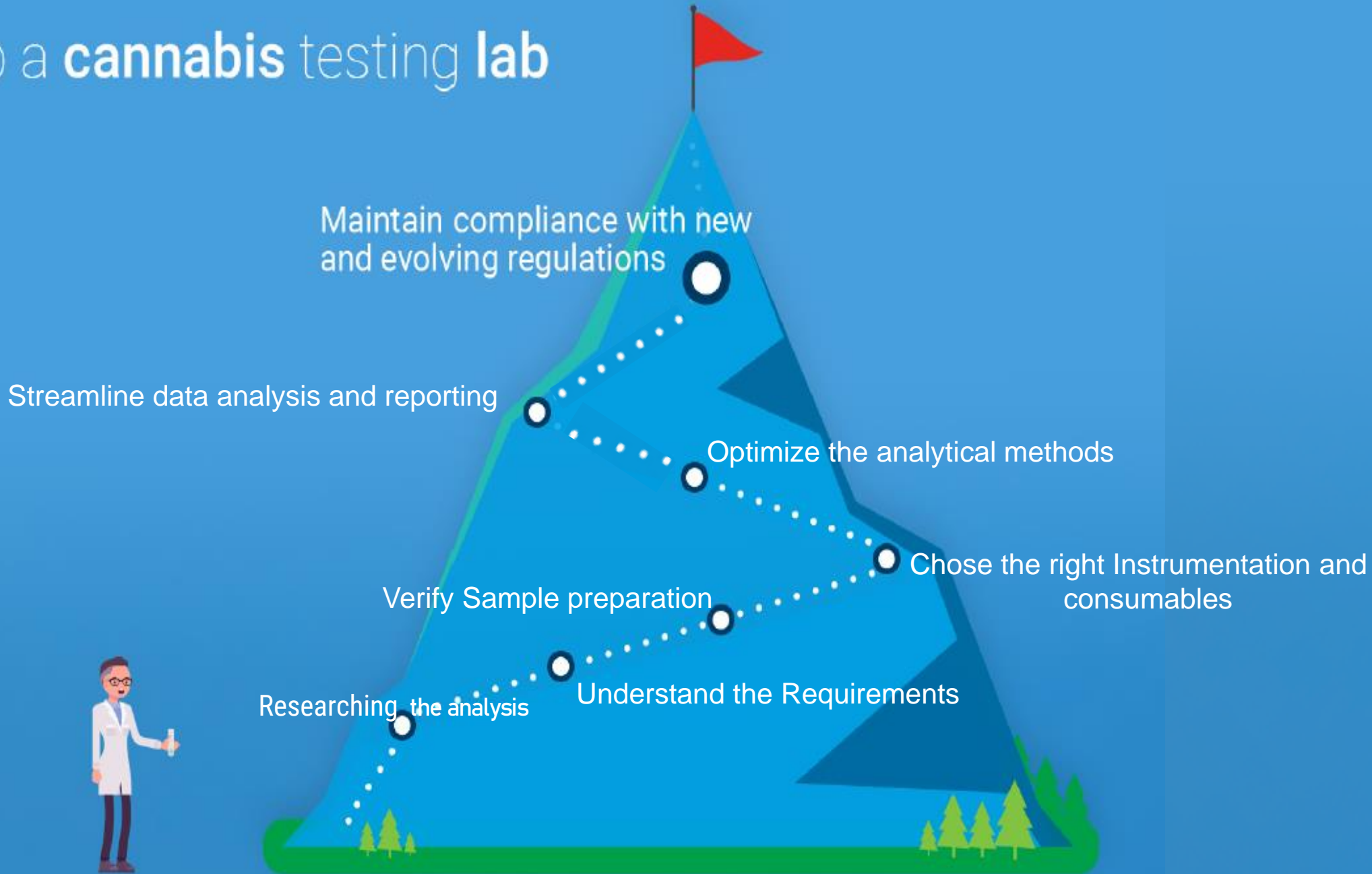


eMethods – 1. What are eMethods

2. Cannabis eMethods for US and Canada



Setting up a cannabis testing lab



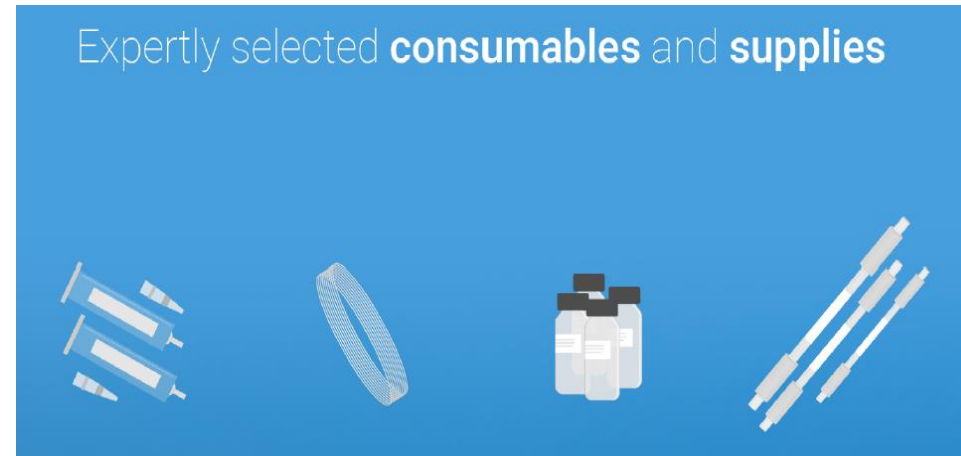
Steps to take before analyzing cannabis

1. Researching the analysis
2. Understanding the requirements



Steps to take before analyzing cannabis

1. Researching the analysis
2. Understanding the requirements
3. **Choosing the right instruments and consumables and supplies**



Steps to take before analyzing cannabis

1. Researching the analysis
2. Understanding the requirements
3. Choosing the right instruments and consumables
4. **Verifying sample preparations**



- Efficient
- Simple and fast
- Cost effective
- Good recoveries
- Easy to reproduce

Steps to take before analyzing cannabis

1. Researching the analysis
2. Understanding the requirements
3. Choosing the right instruments and consumables
4. Verifying sample preparations
5. Optimizing the analytical methods

Compound Name	Precursor Ion (m/z)	Product Ion (m/z)	Fragmentor (V)	Collision Energy (V)	Cell Acceleration (V)	Retention Time (min)	Retention Time Window (min)	Polarity
Acephate	184	143	60	5	4	1.25	1	Positive
Acephate	184	95	60	20	4	1.25	1	Positive
Acequinocyl	402.3	343.2	90	10	4	8.45	1	Positive
Acequinocyl	402.3	189.1	90	41	4	8.45	1	Positive
Acequinocyl	385.3	189.1	90	41	4	8.45	1	Positive
Acetamidrid	223	126.1	100	20	3	3.48	1	Positive

HPLC method conditions

Parameter	Value
Column	Agilent Poroshell 120 phenylhexyl, 3 × 100 mm, 2.7 μm (p/n 695975-312)
Guard Column	Agilent Poroshell 120 phenylhexyl, 3 × 5 mm, 2.7 μm (p/n 821725-914)
Column Temperature	55 °C
Injection Volume	10 μL (with injector program/pretreatment, see Table 2)
Autosampler Temperature	4 °C
Needle Wash	Flushport (100% methanol), 10 seconds
Mobile Phase	A) 5 mM ammonium formate/0.1% formic acid in water B) 0.1% formic acid in methanol
Gradient Flow Rate	0.5 mL/min
Gradient	Time (min) %B
	0.00 30
	1.00 30
	2.00 75
	8.00 96
	9.00 100
	9.50 100
9.51 30	
Analysis And Re-equilibration Time	13 minutes
Total Run Time (Sample to Sample)	13 to 14 minutes

Injector Program/Pretreatment

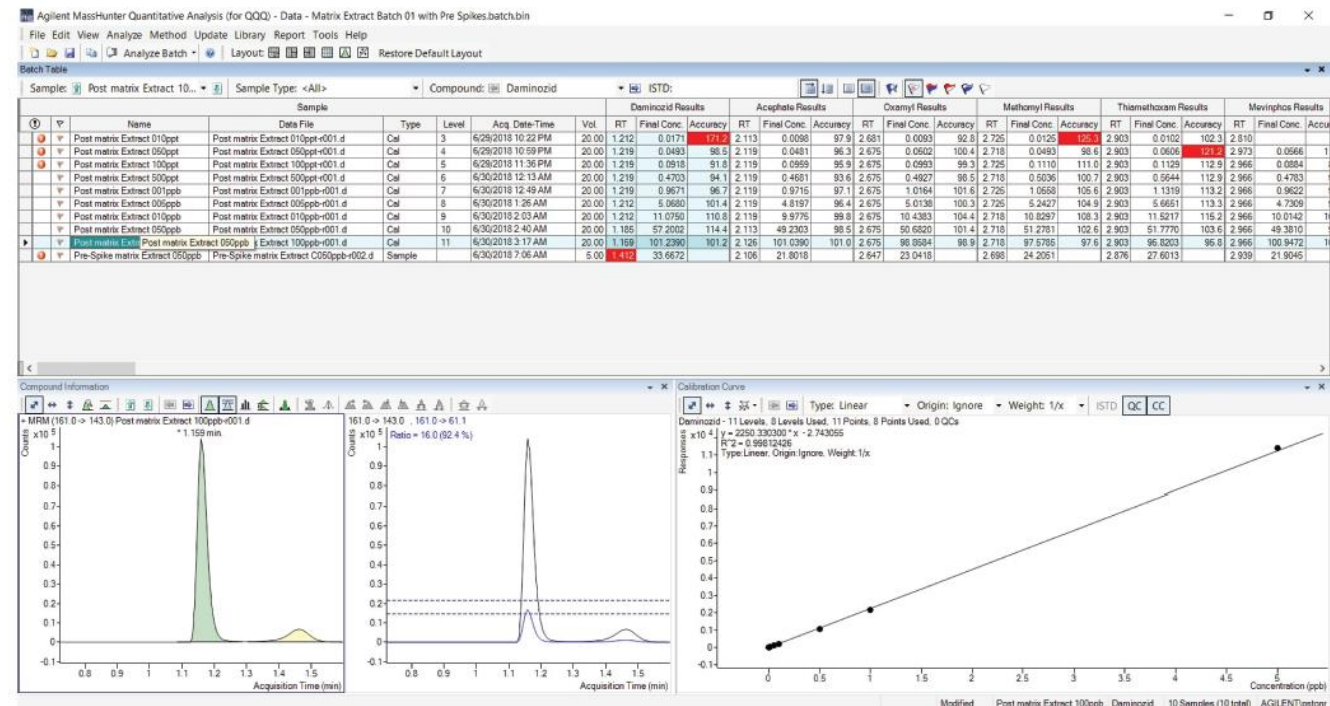
Step	Action	Description
1	Draw	Draw 10 μL from location 1 with default speed using default offset (100 % deionized water)
2	Draw	Draw default volume from the sample with default speed using default offset
3	Wash	Wash needle in flush port for five seconds (100 % methanol)
4	Draw	Draw 10 μL from location 1 with default speed using default offset (100 % deionized water)
5	Mix	Mix 30 μL volume from air with maximum speed five times
6	Inject	Inject

Mass spectrometer configuration and conditions

Parameter	Value
Configuration	6470 or Ultivo triple quadrupole mass spectrometer equipped with Agilent Jet Stream (AJS) ESI source
Ion source conditions	
Ion mode	AJS ESI, positive and negative polarities
Capillary voltage	5,000 V
Drying gas (nitrogen)	13 L/min
Drying gas temperature	200 °C
Nebulizer gas (nitrogen)	55 psi
Sheath gas temperature	200 °C
Sheath gas flow	10 L/min
Nozzle voltage	500 V
Q1 and Q2 resolution	0.7 amu [autotune]
Delta EMV	0 V

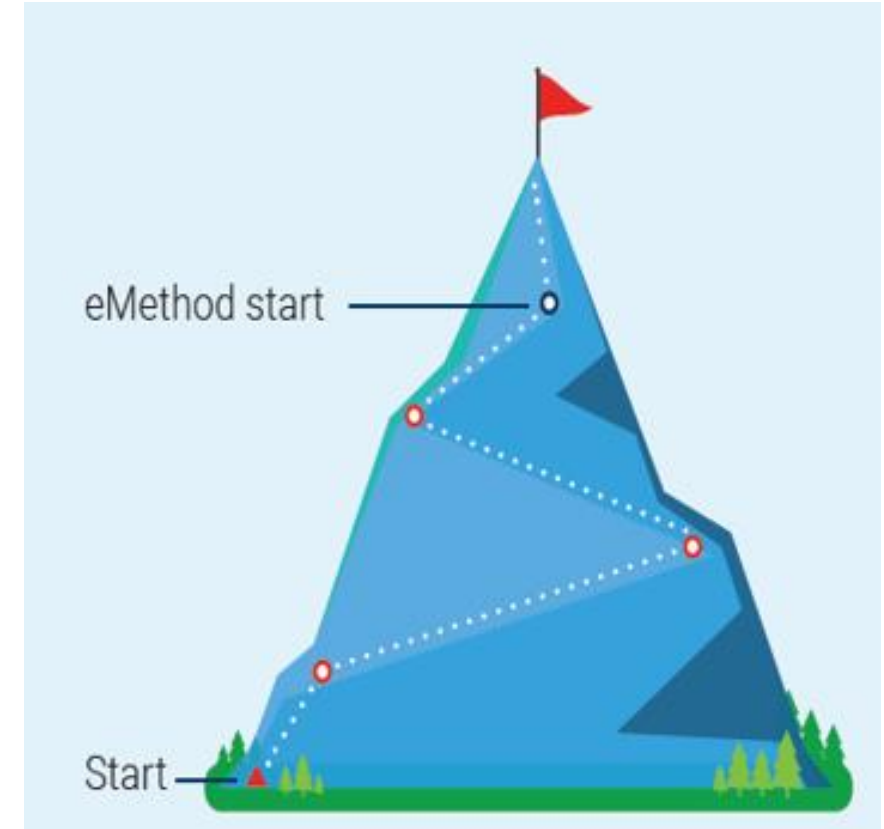
Steps to take before analyzing cannabis

1. Researching the analysis
2. Understanding the requirements
3. Choosing the right instruments and consumables
4. Verifying sample preparations
5. Optimizing the analytical methods
6. Streamlining data analysis and reporting




With eMethods, Agilent has done the hard work for you

1. Researching the analysis
2. Understanding the requirements
3. Choosing the right instruments and consumables
4. Verifying sample preparations
5. Optimizing the analytical methods
6. Streamlining data analysis and reporting




Compared to starting from the ground up, in an eMethod, we have already done most of the initial work for you.

Agilent eMethod Import (MassHunter GC)
✕



eMethod

- Introduction
- eMethod Selection
- Instructions
- Save eMethod
- Finish



Important information, provided by the exporter of this method, is shown below.

Please review this material before continuing. (Click Print to send a copy to a printer.)

Instructions:

1. Instrument Configuration

GC: 8890 GC with Multimode inlet (MMI), mid-column backflush and fast-oven options
MS: 7010B Triple quadrupole detector, EI mode
Autosampler: 7693A Automatic Liquid Sampler Autoinjector and Tray.

2. Software Compatibility

Agilent MassHunter Suite Version 10.0 and greater:

- Agilent MassHunter GC/MS Data Acquisition Version 10, Service Release 1 and greater
- Agilent MassHunter Quantitative Analysis for QQQ Version 10.1 and greater
- Agilent MassHunter Qualitative Analysis for QQQ Version 10 and greater

Optional: Pesticides and Environmental Pollutant 4.0 Enhanced MRM Database. This database allows the expansion the target analyte list. Currently, 27 pesticides are included in the method. The database contains the information on up to 1100 pesticides with up to 8 MRMs per compound.

3. Background Information

Please see application note 5994-1786EN: Analysis of the Twenty-Seven GC-Amenable Pesticides Regulated in the Cannabis Industry in North America with the Agilent 8890/7010B Triple Quadrupole

User:

Instructions:

5. Supporting Files Included with the eMethod

Software file(s)	Direction to user
8890-7010_PestRes Cannabis_GC-TQ MS_acq.M	Acquisition method
8890-7010_PestRes Cannabis_GC-TQ MS.m	Quantitation method
8890-7010_PestRes Cannabis_GC-TQ MS.sequence.xml	Acquisition sequence
23Jan2020_CAL6_Pest_16ppb.D	Example data file

Information files	Description
5994-1786_27 Pesticides in cannabis with 8890GC /7010MS.pdf	Application Note
5994-0973_Optimizing Sample Preparation in Pesticides Analysis for Cannabis	Application Note
5278_010_8890-7010_PestRes Cannabis_GC-TQ MS_Important information.pdf	This document in PDF format

6. Sample Preparation Tips

Matrix:
Dry Cannabis Flower

[Residual Solvents Ordering Guide](#)

5994-4210en

[Terpene Ordering Guide](#)

5994-4211en

[Pesticide Ordering Guide](#)

5994-3517en

Selection and Ordering Information

Residual solvents analysis of cannabinoid products



Intuvo Columns and Supplies

View [MyList](#) for the items in the table below:

Description	Part Number
DB-Select 624 Ultra Inert column (30 m x 0.25 mm x 1.4 µm)**	122-0334UI-HNT
Polyimide gasket, gasket, 5/pk	5190-9072
Intuvo MMI Guard Chip, 2/pk	G4587-60665
Mid column backflush chip	G4588-60721
Flow chip, detector tail, HES MS	G4590-60109
Compression bolt, Intuvo	G4581-60260

**Qty=2 required for setup

GC Columns and Supplies for 7890/8890/8860*

View [MyList](#) for the items in the table below:

Description	Part Number
DB-Select 624 Ultra Inert column (30 m x 0.25 mm x 1.4 µm)**	123-0334UI
Gold plated inlet seal with washer, Ultra Inert, 10/pk	5190-6145

Vials and Caps

View [MyList](#) for the items in the table below:

Description	Part Number
20 mm flat bottom glass crimp top headspace vials, clear, graduation marks and write-on spot	5190-2288
20 mm crimp caps steel/high temperature septa, headspace, 100/pk	5190-3987
20 mm flat bottom glass crimp top headspace vials, amber, graduation marks and write-on spot	5190-2286
20 mm crimp cap, headspace, aluminum, PTFE/silicone septa (100/pk)	5183-4477
20 mm vial crimper	5191-5615
20 mm vial decapper	5191-5613

Standards

View [MyList](#) for the items in the table below:

Description	Part Number
Residual solvent standard cannabis-Universal mix	RSC-100-1
California residual solvent mix 1	SCA-300-1

Cannabis and Hemp Landing Page

Cannabis and Hemp Testing Applications



Potency Testing

Regions that have legalized the use of medicinal or recreational marijuana typically require cannabinoid quantitation for total tetrahydrocannabinol (THC) and cannabidiol (CBD).

[Learn more](#)



Pesticide and Mycotoxin Testing

Potentially harmful pesticides and mycotoxins may be present in cannabis crops and extracts, so analytical monitoring of levels is crucial.

[Learn more](#)



Terpenes Testing

Terpenes contribute to the flavor and fragrance of cannabis. They have also been used to accurately identify and characterize cannabis cultivars.

[Learn more](#)



Heavy Metals and Nutrients Testing

Contamination with heavy metals such as arsenic, lead, cadmium, and mercury pose danger to human health, so cannabis products must be tested for them.

[Learn more](#)



Residual Solvents Testing

Residual solvents are by-products found in processed cannabis. Producers need to incorporate stringent protocols to ensure acceptable levels.

[Learn more](#)



Microbial Testing

The presence of dangerous pathogens in cannabis poses a potential threat to consumers if those microbes synthesize toxigenic mycotoxins.

[Learn more](#)

Terpenes Analysis in Cannabis Products by Liquid Injection using the Agilent Intuvo 9000/5977B GC/MS System
5994-2032EN

Novel Residual Solvents Analysis of Cannabinoid Products with the Agilent Headspace-GC/MS System
5994-1926EN

Analysis of Twenty-Seven GC-Amenable Pesticides Regulated in the Cannabis Industry in North America with the Agilent 8890/7010B Triple Quadrupole GC/MS System
5994-1786EN

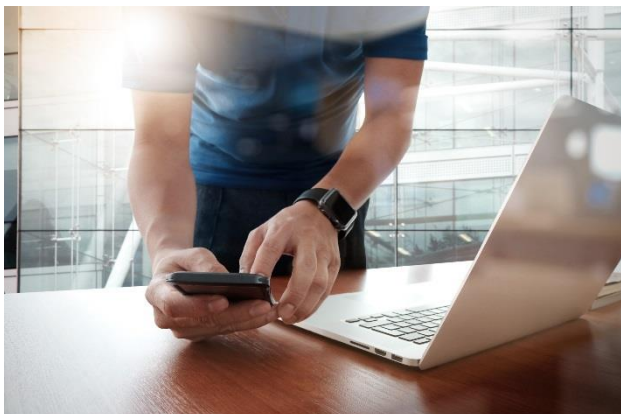
Analysis of Challenging Pesticides Regulated in the Cannabis and Hemp Industry with the Agilent Intuvo 9000-7010 GC/MS/MS system: The Fast-5
5994-1604EN

Pesticides	Residual Solvents	Terpenes
Intuvo	Intuvo	Intuvo
MMI Inlet	MMI or S/SL	MMI or S/SL
ALS	Headspace	ALS
7010 3Q	5977 SQ	5977 SQ
Mid-Column BackFlush 2- HP-5msUI	Mid-Column BackFlush 2-DB-Select 624UI	Mid-Column BackFlush 2-DB-Select 624UI

Questions...



Contact Agilent Chemistries and Supplies Technical Support



1-800-227-9770 Option 3, Option 3:
Option 1 for GC and GC/MS columns and supplies
Option 2 for LC and LC/MS columns and supplies
Option 3 for sample preparation, filtration, and QuEChERS
Option 4 for spectroscopy supplies
Option 5 for chemical standards
Available in the USA and Canada 8–5, all time zones



gc-column-support@agilent.com

lc-column-support@agilent.com

spp-support@agilent.com

spectro-supplies-support@agilent.com

chem-standards-support@agilent.com

Simon_jones@Agilent.com

Agilent CrossLab

From Insight to Outcome

