Introduction

There is a growing need for the analysis of residual solvents and terpenes in cannabis and cannabinoid concentrates. These methods must assure safety and quality and the list of terpenes and residual solvents that are to be analyzed is continuously being increased by state and local governing bodies. A common configuration for these analyses connects headspace sampling techniques with GC or GC-MS systems. Agilent is collaborating with major laboratories to rapidly develop high-end instrument configurations and methodologies that are robust, accurate and precise while still allowing for easy to implement workflows in high productivity laboratories. These methods must assure safety and quality and the list of terpenes and residual solvents that are to be analyzed is continuously being increased by state and local governing bodies. A common configuration for these analyses connects headspace sampling techniques with GC or GC-MS systems. The Agilent headspace-GC-MS systems for the analysis of residual solvents and terpenes includes the 7697A headspace auto-sampler, the 7890B or the Intuvo 9000 GC and the 5977B mass spectrometer operated on MassHunter software. This presentation will discuss the Agilent systems and methodologies for the rapid and robust analysis of terpenes in cannabis and cannabinoid samples.

Experimental

Workflow for samples and standards:
10μL of the a standard is placed in a 10mL headspace vial and capped. A 5 or 10 point calibration curve is created from the standards.

As plant and extract material does not dissolve in solvent, a full evaporation headspace technique (FET) is used for quantitation.

A part of the flower weighing 1.0 gram is frozen, followed by grinding to ensure a representative sample. 10-30 mg of the flower is then weighed into a headspace vial and capped.

For different species of samples:
plant, extracts, etc. Use 5-7 mg +/- 15 mg place into 10 mL headspace vial, evaluate against curve.

Run QC to verify performance

7697A Headspace parameters
- Carrier Control: GC
- Instrument Conditions: GC
- Oven Temperature (°C): 120
- Loop Temperature (°C): 120
- Transfer Line Temperature (°C): 140
- Vial Equilibration (min): 10.00
- Injection Duration (min): 0.50
- GC Cycle Time (min): 10.00
- Vial Size: 10
- Vial Shaking: Level 1

Front SS Inlet He
- Mode: Split
- Heater: 220 °C
- Pressure: 29.45 psi
- Total Flow: 303 mL/min
- Septum Purge Flow: off
- Split Ratio: 100:1

7890B GC Column Summary
- Oven Temperature Program (Initial): 60 °C
- Hold Time: 0.5 min
- #1 Rate: 45 °C/min
- #1 Value: 150 °C
- #1 Hold Time: 0 min
- #2 Rate: 35 °C/min
- #2 Value: 250 °C
- #2 Hold Time: 0.5 min

Front Detector FID Conditions
- Makeup He: 300 °C
- H2 Flow: 40 mL/min
- Air Flow: 400 mL/min
- Makeup Flow: 25 mL/min

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