Poster Reprint

ASMS 2020
WP 174

Characterization of Hemp-Based Products Using HS-GC/MS

Jennifer Sanderson,

Agilent Technologies, Inc. 2850 Centerville Rd, Wilmington, DE 19808

Introduction

The legalization of hemp[1] is driving the partnership between private sector agencies, regulating bodies and the state and federal governments to work together to create common sense guidelines that will provide the framework for methodologies and reporting requirements surrounding the hemp and medicinal cannabis industry. Products made from hemp have and will continue to come under scrutiny not only for the cannabinoid potency, but also for residual solvents, residual pesticides and terpene profiles. Presented here is a complete workflow for residual solvents using headspace gas chromatography-mass spectrometry (HS-GC/MS) for several hemp based consumer products.

Instrumentation

The Agilent 7697A Headspace, was coupled to the 8890/5977B GC/MS The GC was equipped with a Agilent VF-35MS UI column and the MSD with an inert electron ionization (EI) Ion Source and was run in full scan mode .

Figure 1: Agilent 7696A Headspace and 8890 Gas Chromatograph

Experimental

Calibration Preparation

For universal calibration, a saturated brine solution was made by adding 6 grams of sodium chloride to 18.2 MΩ water and shaken rigorously until a cloudy solution formed. With the use of analytical standards listed in Table 1, 5 calibration levels were created by spiking the appropriate aliquot into a 10mL headspace vial containing brine solution to bring each calibrator to a total liquid volume of 3mL. Class I residual solvents calibrator ranged from ~0.15ppm to 50ppm. Class II residual solvents ranged from ~10ppm-1,000ppm.

Analytical Standards	
USP 467 Class 1	USPM-467J-1
USP 467 Class2B	USPM-467-L-1
California Residual Solvent Mix	SCA-300-1

Table 1: Analytical Standards used for calibration and matrix spikes

Sample Preparation

Five replicates of hemp bath ball, hemp cream, hemp gummies and hemp oil were prepared by dissolving 200mg of homogenized sample into 5mL of 18.2 MΩ water, shaken for 2 hours. After the samples were shaken, 500µL of each sample was transferred into 2.5mL of saturated brine solution in a 10mL headspace vial and sealed for analysis.

Matrix Spikes

For each hemp matrix tested, two of the five replicates were spiked with the analytes of interest to determine spike recoveries. This will allow multiple matrices to be analyzed using a single set of calibrators, which will increase sample throughput.

Method Precision

To evaluate the precision of this method, 8 replicate standards were created by spiking the lowest calibrator concentration into brine solution and analyzed. The %RSD was calculated for each analyte.

Results and Discussion

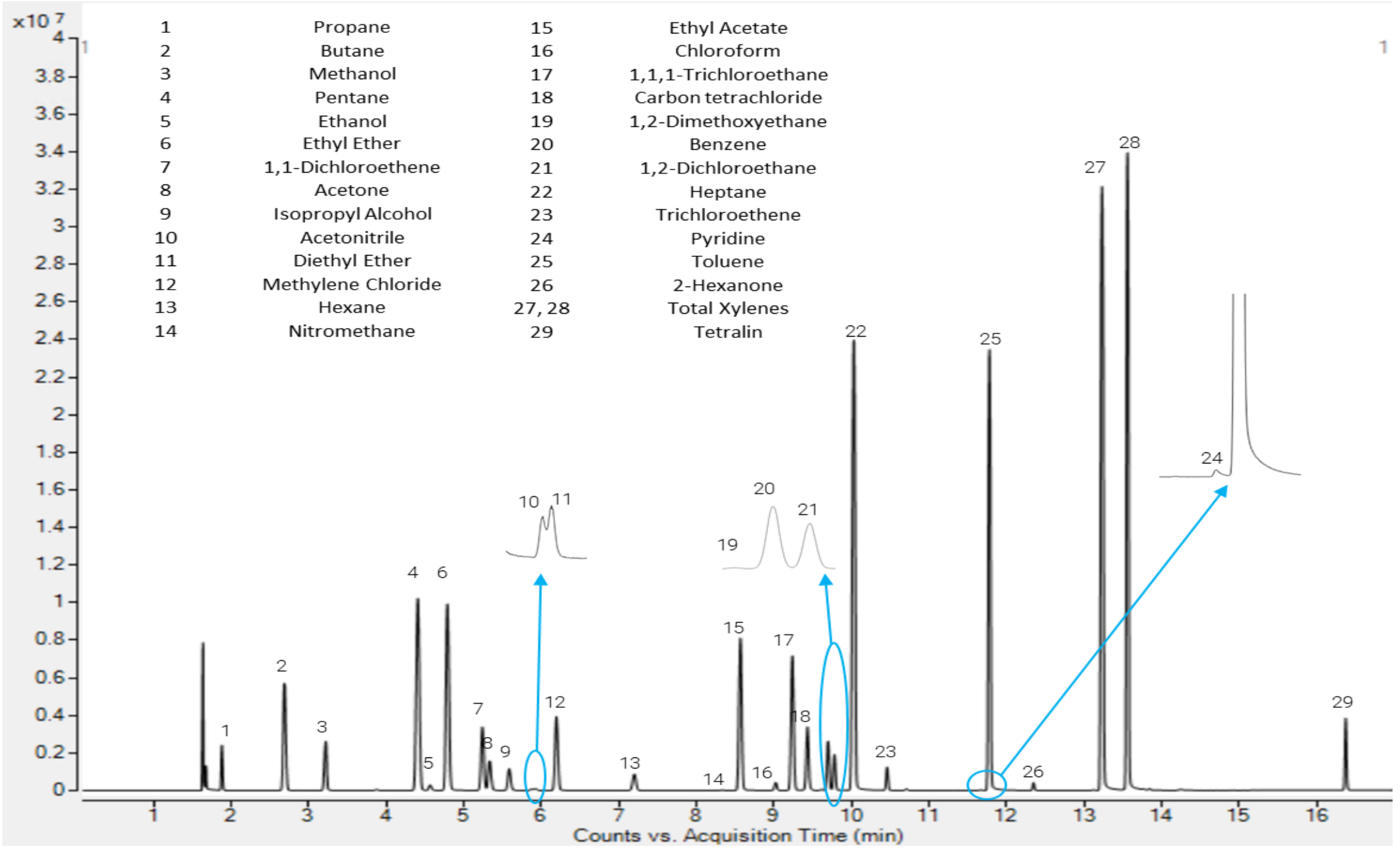
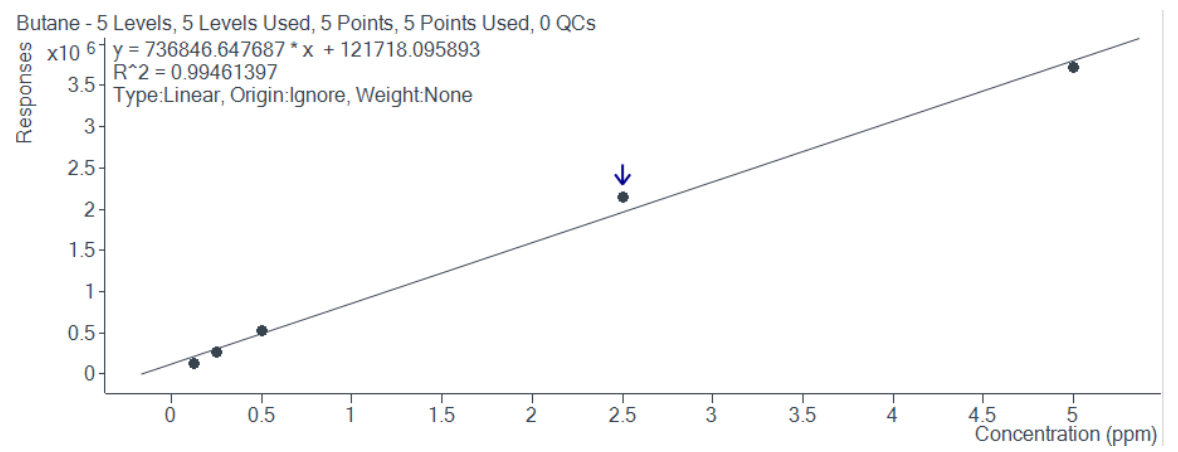
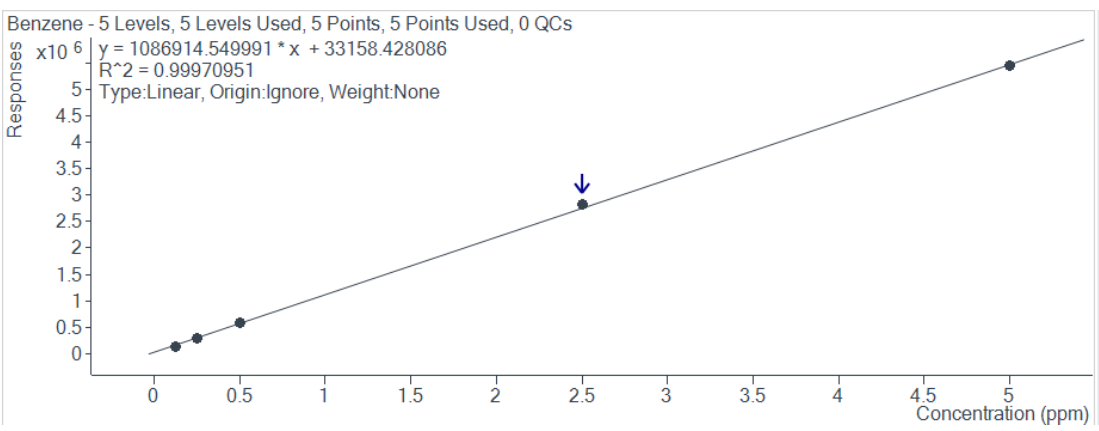


Figure 2: Chromatogram of Residual Solvent Calibrator extracted from brine matrix.

Chromatographic Separation and Linear Calibration

Chromatographic separation of 29 residual solvents is shown in figure 2. With a total sample cycle of 23 minutes, ultra light hydrocarbons, chlorinated solvents, alcohols and nitrogen containing compounds are well resolved with excellent run to run reproducibility. Each of the analytes were analyzed at 5 levels to create linear calibration curves. Sample calibrations can be seen below in figure 3. Reproducibility of the select analytes at the lowest calibrator level is shown in figure 4.





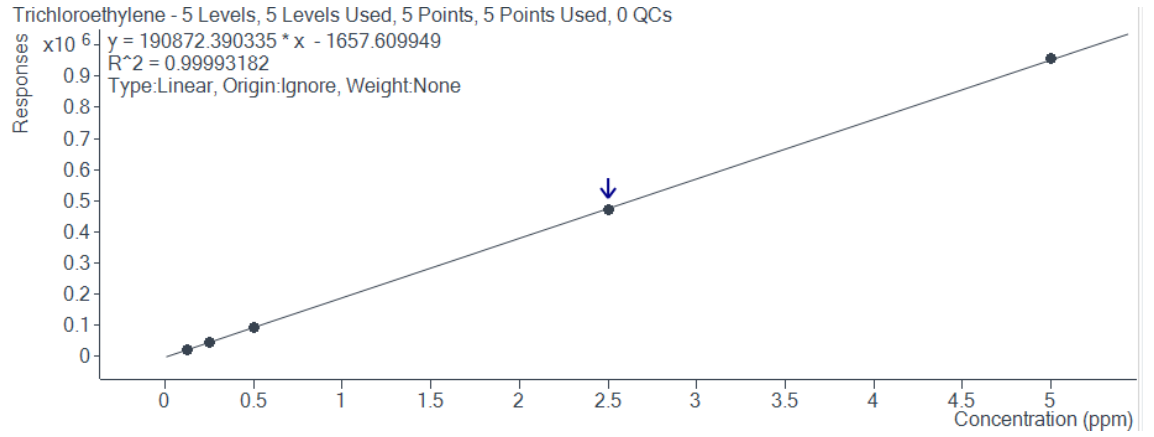


Figure 3: Calibration curves for Butane, Benzene and Trichloroethene

Results and Discussion

Matrix Spike Recoveries

Analyte	Spike Level (ppm)	Hemp Bath Ball Rec. (%)	Hemp Cream Rec. (%)	Hemp Gummies Rec. (%)	Hemp Oil Rec. (%)
Propane	63	110	107	96	88
Butane	63	107	103	96	89
Methanol	75	99	101	95	90
Ethanol	63	90	92	94	86
Isopropyl Alcohol	63	91	93	90	85
Nitromethane	1	92	96	80	75
Ethyl Acetate	63	105	104	102	94
Chloroform	1	99	96	95	79
Benzene	3	102	99	97	79
1,2-Dichloroethane	6	99	96	95	81
Heptane	63	119	114	113	95
Trichloroethene	1	101	96	95	73

Table 2: Matrix Spike Recoveries of select analytes.

Sample Analysis and Matrix Spike Recoveries

The Hemp cream showed trace amounts of isopropyl alcohol that was detectable, but below the LOQ . All other products were negative for all residual solvents calibrated for.

Each of the hemp products were spiked in duplicate to evaluate the recovery of analytes from their respective matrix with a range of 70-130% recovery. Hemp oil showed lower recoveries of all analytes. Hemp cream, bath balls and hemp gummies all had recoveries well within the criteria for all analytes.

Method Precision

Excellent reproducibility was demonstrated by analyzing multiple replicates of the lowest level calibrator. All analytes had %RSD within 10% Figure 4 shows 7 chromatographic overlays of select analytes, along with the %RSD obtained to demonstrate precision.

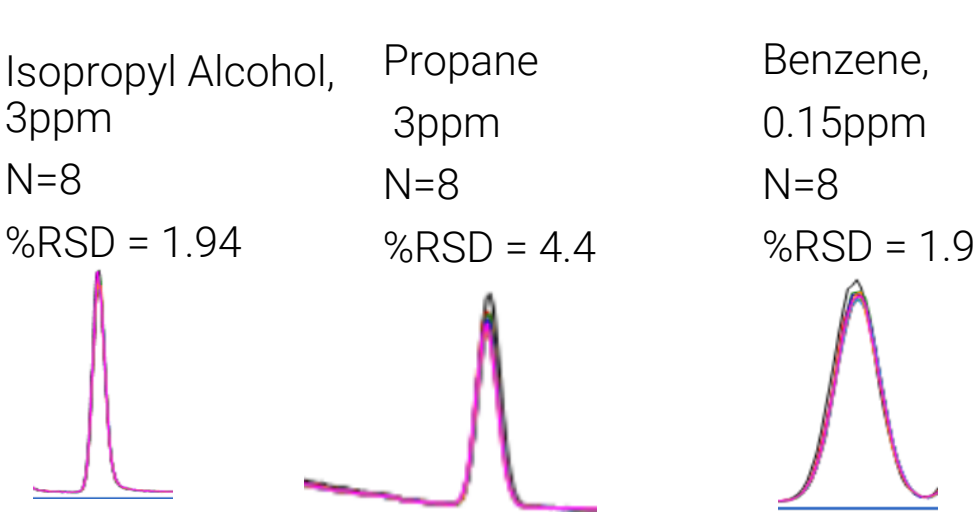


Figure 4: Seven chromatographic overlays for isopropyl alcohol, propane and benzene

Conclusions

Analysis for residual solvents in a variety of hemp consumer products is possible use HS-GC/MS

- Simplified sample extraction and sample preparation makes it easier to evaluate multiple matrices with a single calibration.
- The use of mass spectrometry coupled with headspace gas chromatography allows for identification and quantitation of residual solvents.

References

[1] H.R.2-Agriculture Improvement Act of 2018.n.b. SEC. 10111.

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.

Published in USA, June 1, 2020

Agilent
Trusted Answers