

# Residual Solvent Analysis in Hemp Consumer Products Using Headspace Gas Chromatography and Mass Spectrometry

## Author

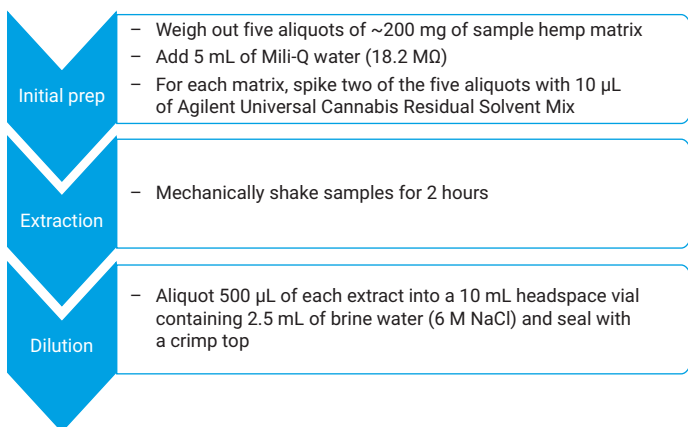
Jennifer Sanderson  
Agilent Technologies, Inc.

## Introduction

The legalization of hemp is driving the partnership between private sector agencies, regulating bodies, and state and federal governments to work together to create common sense guidelines. These guidelines 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 cannabinoid potency, but also for residual solvents and residual pesticides. Presented here is a brief demonstration of the determination of residual solvents in different hemp consumer products using headspace gas chromatography and mass spectrometry.

## Experimental

Hemp bath balls, hemp gummies, hemp oil, and hemp cream of different commercial brands were purchased from an online store to test for residual solvents. Each of these products was prepared using the sample preparation outlined in Figure 1. The analysis was carried out on an Agilent 8890 GC system equipped with an Agilent 7697A headspace sampler and an Agilent 5977B GC/MSD with inert extractor ion source. Method parameters for the instrument are listed in Tables 1 and 2.



**Figure 1.** Sample preparation workflow for preparing the selected consumer products for analysis.

**Table 1.** Headspace sampler method parameters.

Headspace Parameters	Agilent 7697A Headspace Sampler
<b>Temperature Settings</b>	
Oven Temperature	85 °C
Loop Temperature	85 °C
Transfer Line Temperature	100 °C
<b>Timing Settings</b>	
Vial Equilibration	30.00 min
Injection Duration	0.50 min
GC Cycle Time	23.00 min
<b>Vial and Loop Settings</b>	
Vial Size	10 mL
Vial Shaking	Level 8, 188 shakes/min
Fill Mode	Default
Fill Pressure	15 psi
Loop Fill Mode	Custom
Loop Ramp Rate	20 psi/min
Loop Final Pressure	12 psi
Loop Equilibration Time	0.05 min

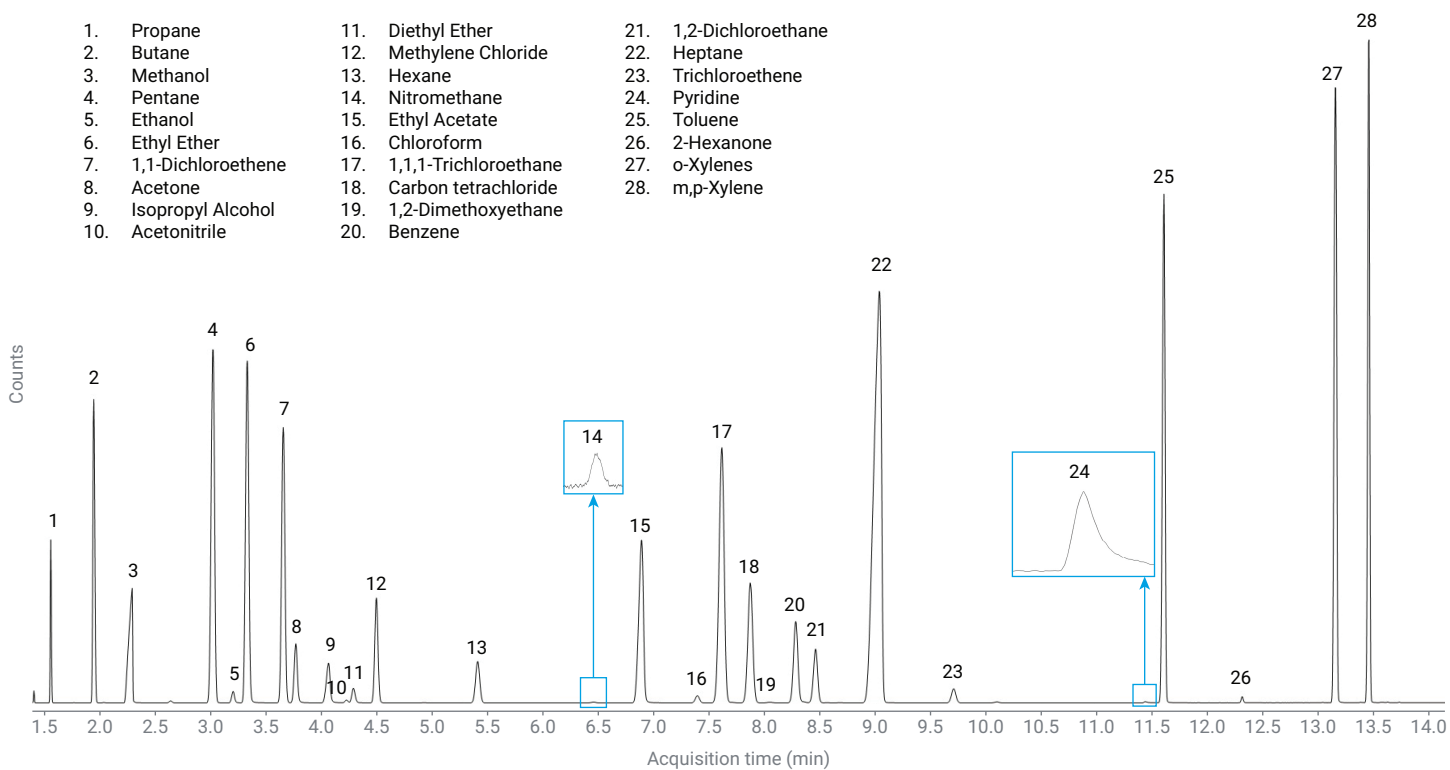
**Table 2.** GC and MSD method parameters.

GC Parameters	Agilent 8890 GC
Liner	Ultra Inert, split, straight
Injection Mode	Pulsed split, 50:1, pulse at 40 psi for 1.5 min
Inlet Temperature	180 °C
Column	Agilent J&W VF-624ms 30 m × 0.25 mm × 1.4 µm
Oven Program	35 °C (hold for 1.5 min), ramp 5 °C/min to 60 °C (hold for 1 min), ramp 20 °C/min to 220 °C (hold for 1.5 min)
Equilibrium Time	0.5 min
Column Flow	Constant, 1.4 mL/min
Transfer Line	220 °C
MSD Parameters	Agilent 5977B GC/MSD
Acquisition Mode	Scan <i>m/z</i> 29 to 250
Solvent Delay	None
Tune File	Atune
Gain	1
MS Source Temperature	230 °C
MS Quadrupole Temperature	150 °C
Duty Cycle Time	23 min

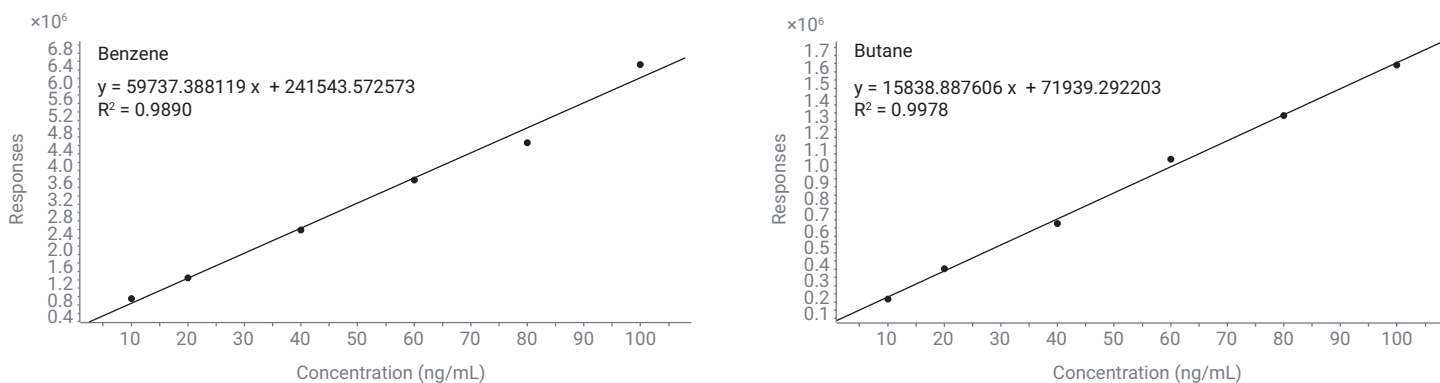
## Results and discussion

A five-point calibration was carried out in brine matrix solution. Figures 2 and 3 show the resultant chromatogram and sample calibration curves. Each of the samples measured against the calibration curve resulted in no residual solvents detected.

Each of the hemp products were analyzed with levels of prespiked residual solvents to evaluate recovery from the various matrices. Spike levels and recoveries are listed in Table 3. The bath balls, gummies, and cream all had recoveries of the various residual solvents within 90 to 110%, except for heptane, which consistently recovered high across the matrices. Hemp oil had notably lower recoveries of prespiked residual solvents.



**Figure 2.** Total ion chromatogram (TIC) of the Agilent Universal Cannabis Residual Solvent Mix in brine water matrix. The saturated salt matrix allows for alcohols and other less volatile solvents to go into the headspace.



**Figure 3.** Five-point calibration curve for residual solvents butane (625 to 6,250 ppm) and benzene (0.5 to 5 ppm) in brine matrix.

**Table 3.** Prespiked consumer hemp products tested for recovery of residual solvents.

Matrix Spike Recoveries					
Analyte	Spike Level (ppm)	Hemp Bath Ball Recovery (%)	Hemp Cream Recovery (%)	Hemp Gummies Recovery (%)	Hemp Oil Recovery (%)
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

## Conclusion

A simple aqueous extract of several hemp-based consumer products was evaluated using Agilent chemical standards to calibrate and prespike the matrices to quantitate residual solvents.

## Appendix

**Table 4.** Chemical standards referenced in this application.

Part Number	Agilent Cannabis Residual Solvent Standards
RSC-CBS-CA	California Cannabis Residual Solvent Kit (in triacetin)
RSC-CBS-CO	Colorado Cannabis Residual Solvent Kit (in DMSO)
RSC-CBS-CAN	Canada Cannabis Residual Solvent Mix (in triacetin)
RSC-CBS-UNI	Universal Cannabis Residual Solvent Mix (in triacetin)

To see the complete lists of lab supplies for cannabis and hemp workflows, please visit [www.agilent.com/chem/cannabis-workflow-ordering-guide](http://www.agilent.com/chem/cannabis-workflow-ordering-guide).

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