

Accelerated Solvent Extraction of Terpenes in Cannabis Coupled with Various Injection Techniques for GC-MS Analysis

Myers, Colton¹; Herrington, Jason S.¹; Hamrah, Paul²; Anderson, Kelsey²

1. Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823, United States
2. Verity Analytics, 8888 Miramar Road, San Diego, CA 92126, United States

Supplementary Material

Table S1 - Analytical parameters for evaluating terpenes in hops with HS-Syringe, HS-SPME Arrow, and DI-SPME Arrow.

Agilent 7890B/5977B GC-MS Parameters	
Column	Rxi-624Sil MS – 30m x 0.25mm x 1.4µm (cat# 13868)
Injection	HS-Syringe in Error! Reference source not found. HS-SPME Arrow and DI-SPME Arrow in Error! Reference source not found.
Mode	Split (50:1)
Liner	Topaz 1.8 mm ID Straight/SPME Inlet Liner (cat# 23280)
Inj. Temp.	280 °C
Purge Flow	3 mL/min
Oven	55 °C (hold 0 min) to 130 °C (hold 6 min) by 25 °C/min to 280 °C (hold 1 min) by 30 °C/min
Carrier Gas	He, constant flow
Flow Rate	2.0 mL/min
Detector	HES - MS
Mode	Scan
Transfer Line Temp.	280 °C
Source Temp.	350 °C
Quad Temp.	200 °C
Acquisition Range	35 - 350 amu
Rate	8.2 scans/sec
Solvent Delay	3.00 min

Table S2 - HS-Syringe parameters for terpenes introduction to GC-MS.

CTC PAL Parameters	
Tool	HS-Syringe
Syringe Temperature	150 °C
Agitator	Agitator 1
Injector Penetration Depth	40 mm
Incubation Time	30 min
Incubation Temperature	80 °C
Purge	90 s

Table S3 - HS-SPME Arrow and DI-SPME Arrow parameters for terpenes introduction to GC-MS.

CTC PAL Parameters	
Tool	HS-SPME / DI-SPME
Agitator	Agitator 1
Heatex Stirrer	Heatex Stirrer 1
Injector Penetration Depth	50 mm
Incubation Time	2 min
Extraction Time	4 min
Incubation / Extraction Temperature	40 °C
Desorption Time	60 s
Pre / Post Conditioning	Yes / No
Conditioning Time	60 s
Conditioning Temperature	280 °C

Table S4 - MS SIM Parameters.

#	Name	Retention Time (min)	Masses
1	α -Pinene	4.52	91, 92.1, 93.1
2	Camphene	4.88	79.1, 93.1, 121
3	β -Myrcene	5.2	93.1
4	β -Pinene	5.39	79.1, 93.1
5	Carene	5.81	77, 93.1
6	α -Terpinene	5.99	77, 93.1
7	<i>trans</i> -Ocimene	6.12	93.1
8	D-Limonene	6.23	93.1
9	<i>p</i> -Cymene	6.33	119
10	<i>cis</i> -Ocimene	6.44	93.1
11	Eucalyptol (1,8-Cineole)	6.57	81
12	γ -Terpinene	6.87	91, 93.1
13	Terpinolene	7.49	91, 93.1
14	Linalool	8.16	91. 92.1, 93.1

15	Isopulegol	9.19	67, 69.1
16	Naphthalene-d8 (ISTD)	9.83	136
17	Geraniol	10.51	67.1, 69.1
18	β -Caryophyllene	12.19	91, 93.1
19	α -Humulene	12.56	91, 92.1, 93.1
20	<i>cis</i> -Nerolidol	13.15	79.1, 91, 93.1
21	<i>trans</i> -Nerolidol	13.42	79.1, 91, 93.1
22	Guaiol	13.95	93.1, 161
23	(-)-Caryophyllene Oxide	14.12	69.1, 79.1, 93.1
24	α -Bisabolol	14.52	93.1, 119

Table S5 - Terpene calibration levels for DI-SPME Arrow (post ASE extraction).

Calibration Curve Serial Dilution Procedure				
Cal Level	Concentration ($\mu\text{g/mL}$)	Amount of previous standard added (μL)	Amount of IPA added (μL)	Final Volume (mL)
7	12.8	--	--	3
6	6.4	1500	1500	3
5	3.2	1500	1500	3
4	1.6	1500	1500	3
3	0.8	1500	1500	3
2	0.4	1500	1500	3
1	0.2	1500	1500	3

Table S6 - Terpene calibration levels for DI-SPME Arrow (post dilution in headspace vial).

Calibrator Concentration at Instrument				
Cal Level	Concentration ($\mu\text{g/mL}$)	Concentration at Instrument ($\mu\text{g/mL}$)	Amount of previous calibrator added (mL)	Final Volume (mL)
7	12.8	0.64	1	20
6	6.4	0.32	1	20
5	3.2	0.16	1	20
4	1.6	0.08	1	20
3	0.8	0.04	1	20
2	0.4	0.02	1	20
1	0.2	0.01	1	20

Table S7 - Terpene calibration levels for LI-Syringe (post ASE extraction and dilution).

Cal Level	Concentration (µg/mL)	Amount of previous standard (µL)	Amount of IPA (µL)	Final Amount (µL)
10	5.12	--	--	500
9	2.56	500	500	500
8	1.28	500	500	500
7	0.64	500	500	500
6	0.32	500	500	500
5	0.16	500	500	500
4	0.08	500	500	500
3	0.04	500	500	500
2	0.02	500	500	500
1	0.01	500	500	1000