APPLICATION BRIEF 22001

Terpenes and residual solvents in hemp products by GC-FID

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Introduction

The production of hemp extracts and concentrates requires the use of solvents such as ethanol, butane or propane. The presence of these solvents in final consumer products must be monitored for safety and quality purposes.

Terpenes are the compounds that provide the unique aroma of hemp and hemp products and are thought to impact the user's experience. Although not typically regulated, terpene profiles can provide valuable information for product quality.

This protocol provides a starting point for the development of a method for detection and quantitation of terpenes and residual solvents in hemp and hemp products for GC analysis with headspace sampling.

Important notes

- This application brief provides starting conditions for residual solvents and terpenes on the Thermo Scientific™ TraceGOLD™ TG-624SilMS GC column, allowing for users to optimize their chromatography for desired target analytes and limits, depending on the local rules and regulations
- For plant material and many oils, concentrates and extracts, a small aliquot (for example, 10 mg or 10 µL) may be placed directly into the headspace vial. If needed, samples may be dissolved in dimethylacetamide (DMA) or another appropriate solvent before analysis.
- The high thermal stability of the TraceGOLD TG-624SilMS GC columns allow for consistent chromatography of late-eluting terpenes

Materials required

- GC-FID instrument such as Thermo Scientific™ TRACE™ 1310 Gas Chromatograph with headspace autosampler
- TraceGOLD TG-624SilMS GC Column, 60 m × 0.32 mm ID × 1.8 µm (P/N 26059-3410)

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Protocol

1. Analyze samples using the following GC conditions:

Headspace parameters	Incubation: 80 °C for 2.5 min
	Syringe temp: 90 °C
	Injection volume: 0.7 mL, Filling vol.: 0.8 mL
	Purge: N_2, 5 s pre-injection, 60 s post-injection
Inlet module mode	SSL, Split with cyclosplitter inlet liner
Split ratio	10:1
Inlet temperature	225 °C
Carrier gas, mode, flow	Helium, 3.25 mL/min constant flow
Oven program	40 °C initial, hold 0.2 min
	17.5 °C/min to 90 °C, hold 1.5 min
	17.5 °C/min to 310 °C, hold 1.37 min
	Total run time of 18.5 min
FID temperature	310 °C

2. Most residual solvents elute in the first half of the chromatogram. If these are the only compounds of interest the run can be shortened.

3. Terpenes begin to elute at approximately 10 minutes in this method. A higher initial temperature can be used for analysis of terpenes only.

Residual solvents and terpenes RTs

Compound	Approximate retention time (min)
Methanol	2.82
Ethanol	3.27
nC5 (Pentane)	3.30
Acetone	3.58
IPA (2-Propanol)	3.62
Acetonitrile	3.78
Carbon Disulfide	3.82
DCM (Methylene Chloride)	3.92
MTBE (2-methoxy-2-methyl propane)	4.09
nC6 (Hexane)	4.31
MEK (2-Butanone)	4.81
2-Butanol	4.89
THF (Tetrahydrofuran)	5.09
Chloroform	5.12
Benzene	5.62
nC7 (Heptane)	5.78
1-Butanol	5.89
Trichloroethylene	6.14
n-Butyl Formate	6.60
Pyridine	7.16
Toluene	7.30
Ethylbenzene	8.72
m+p-Xylene	8.84
o-Xylene	9.21
DMSO	9.26
DMA (n,n-Dimethylacetamide)	9.40
a-Pinene	9.57
Camphene	9.87
ß-Pinene	10.23

Compound	Approximate retention time (min)
a-Phellandrene	10.47
a-Terpinene	10.61
d-Limonene	10.74
p-Cymene	10.77
Sabiene	10.85
Eucalyptol (1,8-Cineole)	10.92
Terpinene	11.03
nC11	11.22
Terpinolene	11.37
Linalool	11.67
(-)-Fenchone	11.88
a-Thujone	12.07
beta-Thujone	12.18
nC12	12.23
dl-Menthol	12.67
nC13	13.16
Nerol (Geraniol)	13.22
Citral-Z (Neral)	13.55
Sulfolane	13.90
nC14	14.01
nC15	14.81
ß-Caryophyllene (ß-Humulene)	14.84
a-Humulene	15.13
nC16	15.56
nC17	16.27
Humulene Epoxide	16.61
a-Bisabolol	16.74
nC18	16.93

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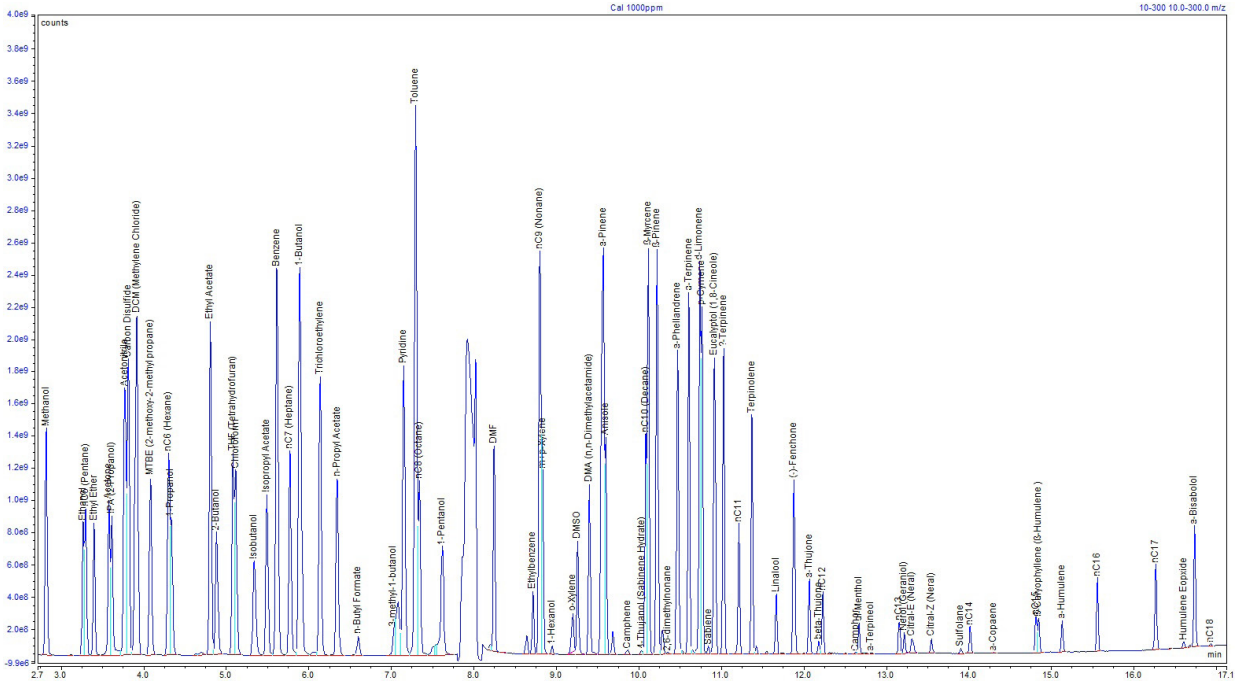

Related products

Description	Part number
TraceGOLD TG-624SilMS column, 60 m × 0.32 mm ID × 1.8 µm	26059-3410
Amber headspace glass vial with crimp cap, 10 mL	10-CV(A)
Headspace glass vial with blue magnetic crimp cap with seal	20-MCBC-ST3

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