Application Note No. 084

In-Liner Derivatisation and LVI-GC-MS of THC in Human Hair

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Introduction

Recently, the analysis of drugs of abuse in human hair has received much attention, primarily as it allows for the determination of long-term trends in drug usage. The analysis of delta-9-tetrahydrocannabinol (THC), the active ingredient of cannabis, and one of its human metabolites 11-nor-delta-9-THC-COOH (THC-COOH) in human hair currently requires solvent extraction of a quantity of hair, concentration of the extract by SPE, derivatisation with BSTFA followed by GC/MS analysis. Using large volume injection with in-liner derivatisation reduces sample preparation and lowers the detection limits.

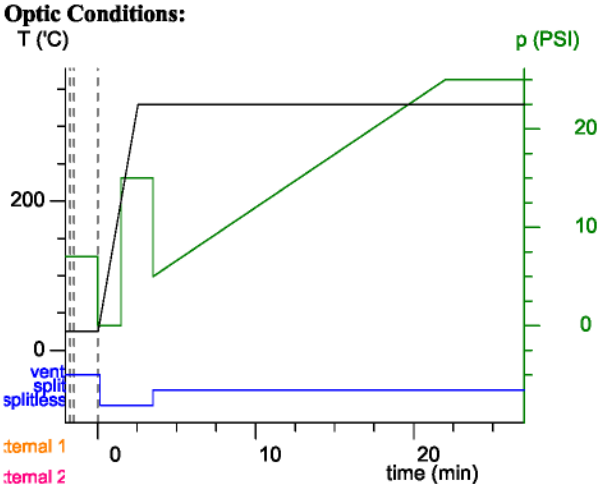
Procedure

1. Inject 125 µL of sample extract in ethyl acetate
2. Vent solvent at initial temperature and purge pressure
3. Inject 2 µL of BSTFA under static flow conditions
4. Heat injector to final temperature for derivatisation
5. Apply pressure to transfer derivatised sample from the injector onto the column in splitless mode
6. Analyse components with pressure ramp and open split line

Instrumentation & Conditions

- ATAS Optic 2-200 programmable injector
- Agilent 6890 with 5973 MSD

Optic Conditions:



Liner:	Packed
Mode:	Expert
Flows: Vent:	100 mL/min
Split:	50 mL/min
Equilibration time:	0:30 m:s 25
Initial temperature:	°C
Ramp rate:	2 °C/s
Final temperature:	330 °C
Vent time:	1.5 mins
Splitless time: Purge	3.5 mins
pressure: Derivatisation	7 psi
pressure: Derivatisation	0 psi
time: Transfer pressure:	1.5 mins
Transfer time: Initial	15 psi
pressure: Final pressure:	2 mins
	5 psi
GC conditions:	25 psi
Column: SGE BP 1 50 m x	
Initial Temperature: Initial	
Time:	0.32 mm i.d. x 0.25 um film
Ramp Rate:	80 °C
Final Temperature:	4 mins
	10 °C/min
	260 °C (5 mins)

MSD conditions:

Mode:		SIM
Ions:	THC:	371, 386, 303
	THC-COOH:	371, 473, 488

Conclusions

The in-liner derivatisation of THC and its metabolites is possible when using the Optic 2 programmable injector in expert mode. A programmable autosampler is necessary to enable the multiple injection of firstly sample extract and then derivatisation agent.

Acknowledgements

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Results

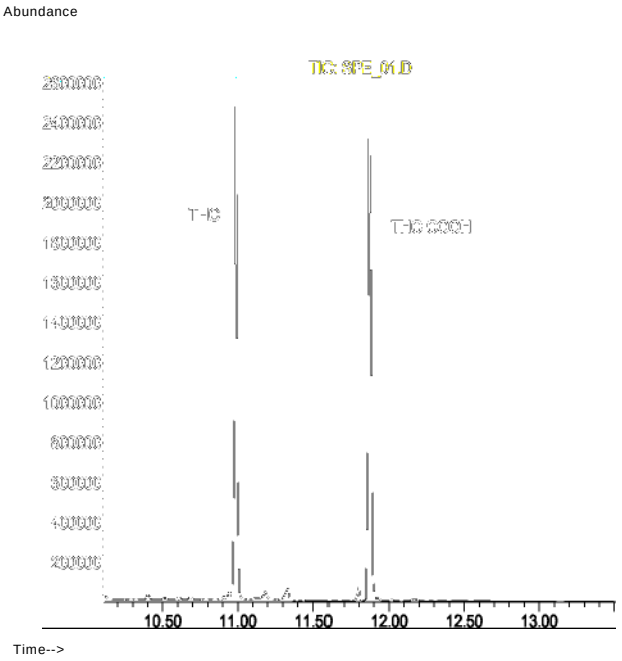

Figure 1: *1 µL splitless injection of a pre-derivatised standard solution*

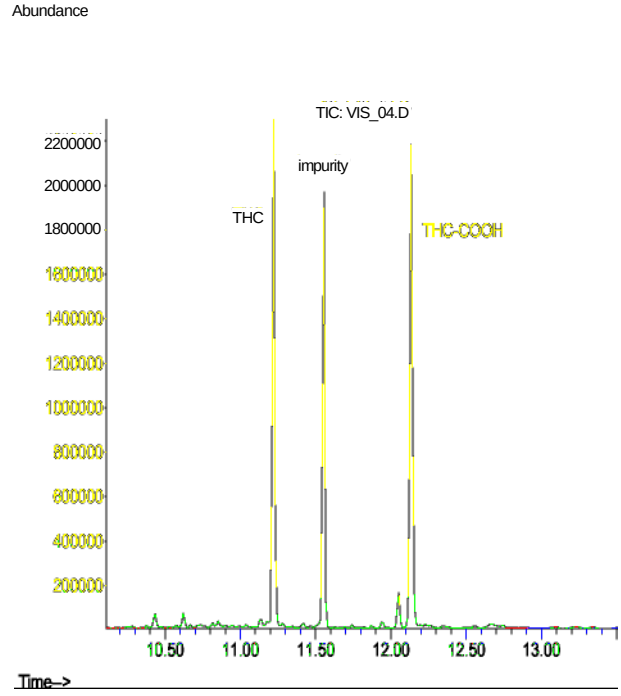

Figure 2: *125 µL large volume injection of standard solution with in-liner derivatisation*

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