

Application Note No. 084

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

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Introduction

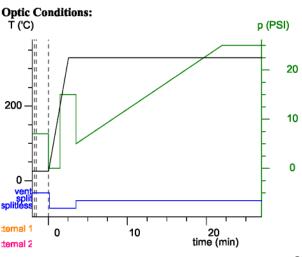
Recently, the analysis of drugs of abuse in human hair has received much attention, primarily as it allows for the determination of long-term trends in drug usage. The analysis delta-9-tetrahydrocannabinol (THC), the active ingredient of metabolites human cannabis. and one of its 11-nor-delta-9-THC-COOH (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 μ L 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



Liner: Packed Mode: Expert Flows: Vent: 100 mL/min Split: 50 mL/min Equilibration time: 0:30 m:s 25 °C Initial temperature: Ramp rate: 2 °C/s Final temperature: 330 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

We would like to thank Gian Marco Currado from LGC for his kind permission to publish this data and Nash Vadukul, LGC for his help in collecting the information.



Results

Abundance TIC: SPE_04.D 2500000 2400000 2201016 200000 THE SECT 1800000 1300000 1400000 1200000 1000000 anne \$0000C 40000C ZULULU 11.00 10.50 12.00 12.50 13.00

Figure 1: 1 μ L splitless injection of a pre-derivatised standard solution

Abundance

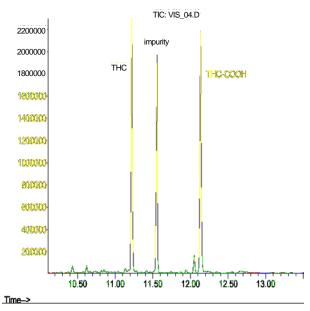


Figure 2: $125~\mu L$ large volume injection of standard solution with in-liner derivatisation