

GC/MS Application Note



CLINICAL



LIFE SCIENCE

Automated Determination of
Drugs of Abuse in Hair Samples





Automated Determination of Drugs of Abuse in Hair Samples

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A fully automated sample preparation method has been developed and tested for extraction and analysis of drugs of abuse from hair samples. Performance was tested using hair samples spiked with cannabinoid standards. The automated sample preparation instrumentation can be directly coupled to any analytical instrumentation for fully automated sample preparation and analysis.

Introduction

Hair testing is a powerful tool routinely used for the detection of drugs of abuse in forensic and toxicology applications. The analysis of hair can be highly advantageous as it can provide prolonged detection, is non-invasive and provides chronological information about drug intake or chemical exposure, allowing patterns of drug abuse and abstinence to be established, in contrast to the analysis of body fluids. The window of detection is dramatically extended to weeks, months or even years when testing hair, and methods for evading urinalysis do not affect hair analysis. The advantages of hair analysis are steadily gaining recognition, as can be seen from its growing use in the following areas of application:

- Evidence of drug abuse
- Gestational drug exposure
- Child Protection
- Workplace drug testing
- Therapeutic monitoring
- Driving Licence Renewal
- Long term poisoning
- Doping Control (sports)
- Stress monitoring
- Drug Facilitated Sexual Assault Investigations
- Environmental Monitoring

However, current methodology routinely involves complex and time-consuming washing, homogenization, derivatisation, sample clean-up techniques and extraction techniques before analysis by GC/LC-MS(/MS). This is the driver for the development of automated sample preparation workflows which include direct coupling to the analytical instrumentation, providing fully automated sample preparation and analysis.

Automation Workflow

A typical manual workflow for hair analysis consists of the following steps:

1. Segmentation (cutting) hair sample
2. Weighing of sample
3. Decontamination (washing)
4. Digestion
5. Extraction
6. Derivatisation
7. Injection, separation and MS analysis
8. Interpretation of results

In this workflow, we can readily automate from step 3 onwards, and the automated workflow for steps 3-7 is shown beneath in figure 1:

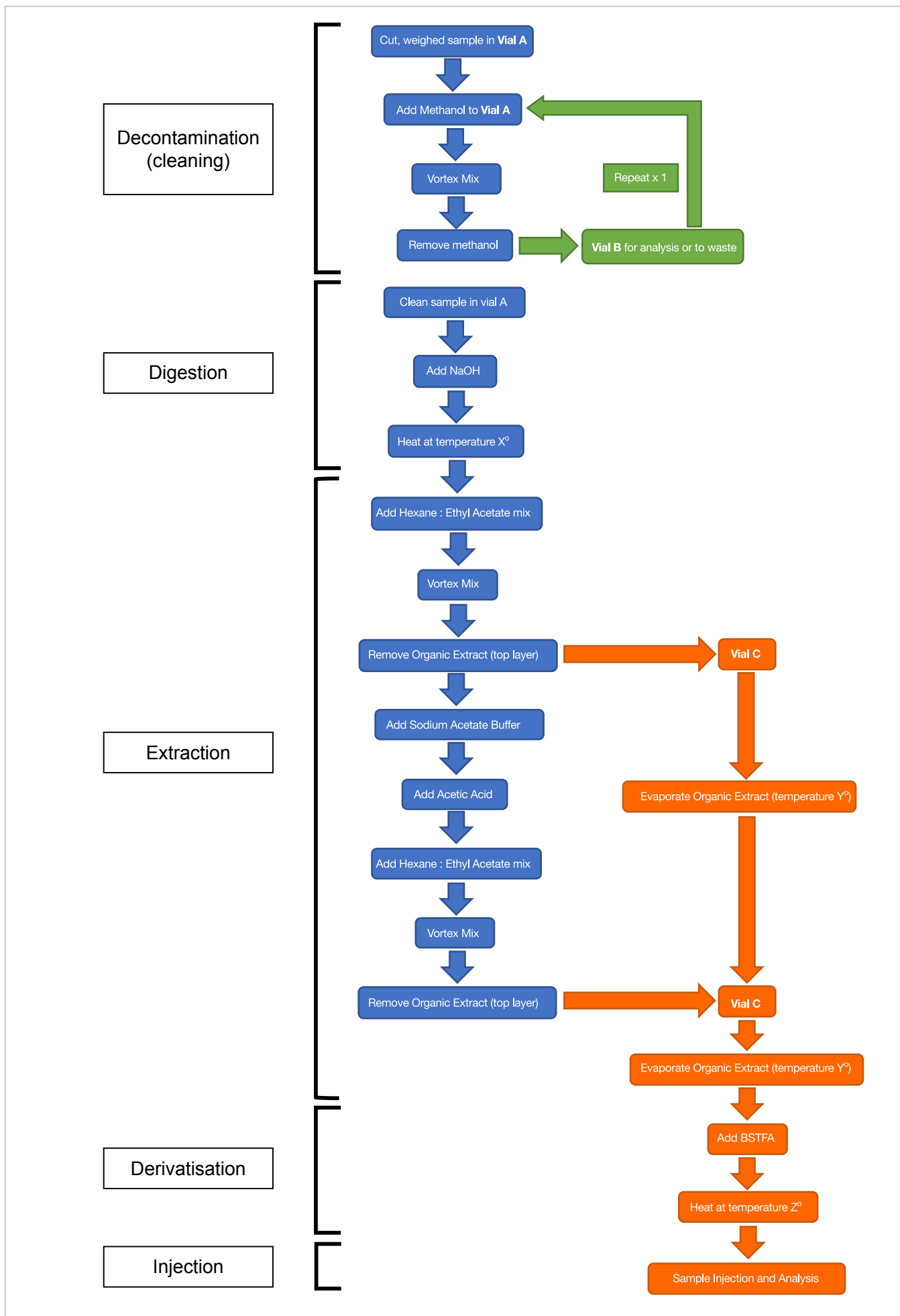


Fig. 1: Automated Workflow steps 3 to 7.

Instrumentation

The instrument used for this automation was a PAL RTC as shown in figure 2.

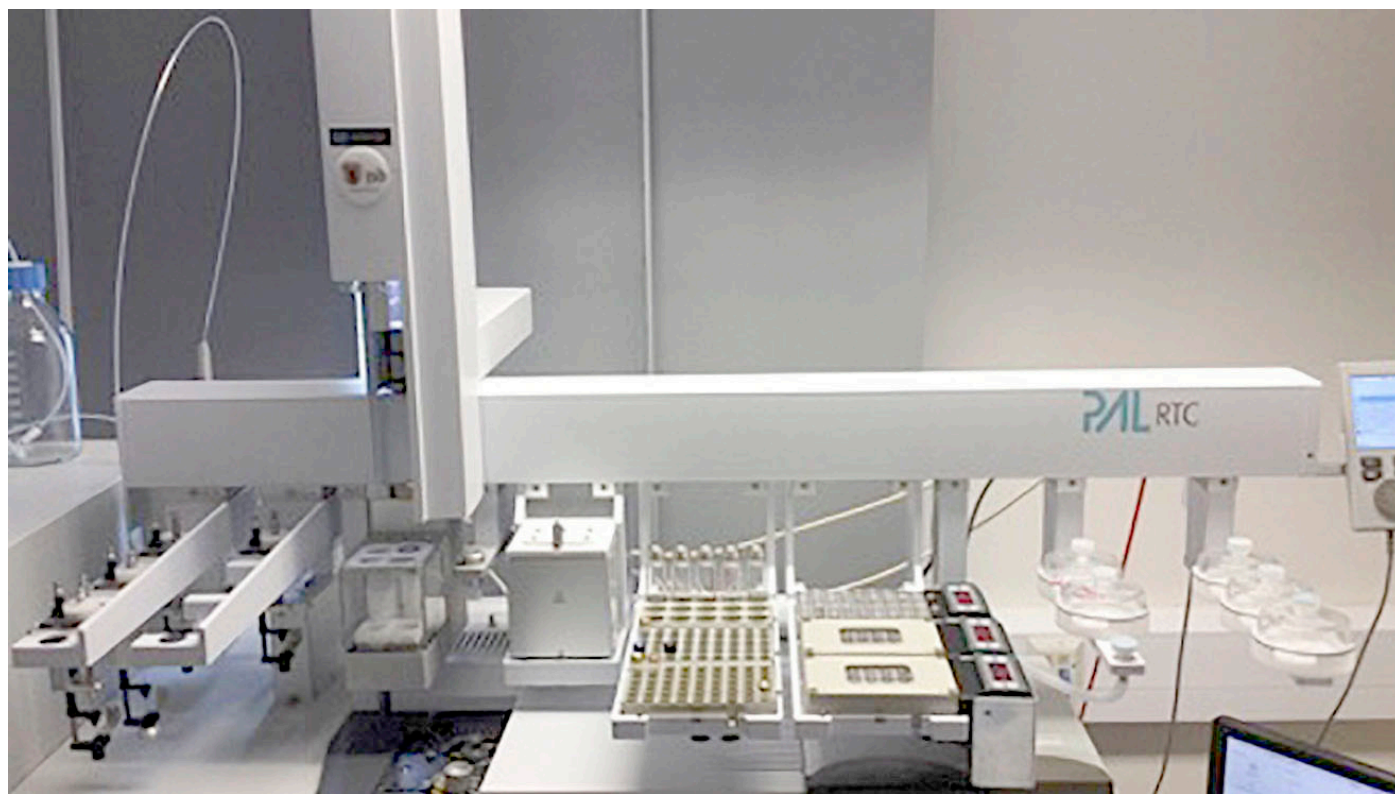


Fig. 2: PAL RTC Configuration as used for the automation workflow.

From left to right in figure 2 (above):

- Dilutor tool (dilutor syringe and valve obscured in this image by the crossrail)
- 2 x park stations to hold a dilutor tool, a headspace tool and 3 x liquid syringe tools to hold syringes of different sizes
- Vortex Mixer
- MHE Adapter
- Heater/Agitator
- Sample tray holder with 1 x VT15 rack holding 20ml vials and 2 x VT54 racks to hold 2ml vials
- Sample tray holder with 1 x HT54-2 heated rack for 2ml vials and 2 x HT12-2 racks for 2ml vials
- Large Wash Module
- Solvent Module

As shown in figure 1, three temperature zones are required for this automation: temperature X during the digestion step, temperature Y during the extraction step, and temperature Z during the derivatisation step. Two of the sample evaporation steps were obstructively time-consuming when carried out at ambient temperature, necessitating heating during evaporation. Due to the number of modules required to achieve this workflow, there was limited space on the crossrail to accommodate 3 heater/agitators in order to provide three heated zones as can be seen in figure 2. Solvent evaporation was achieved using the MHE adaptor, which cannot be used in conjunction with the agitator due to the needle lengths involved. Therefore, in order to optimise this automated workflow, JSB developed heated racks which allow the use of the headspace tool and MHE adaptor in combination with heat, significantly reducing the time taken for solvent evaporation. Examples of these racks are shown in figure 3 below.



Fig. 3: Heated racks, developed by JSB.

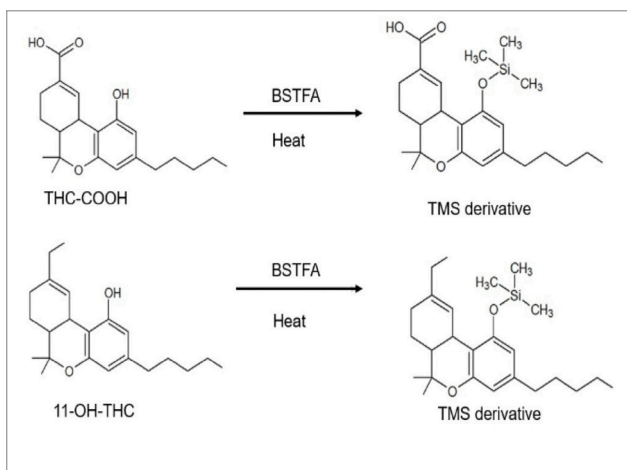


Fig. 4: Derivatization of THC

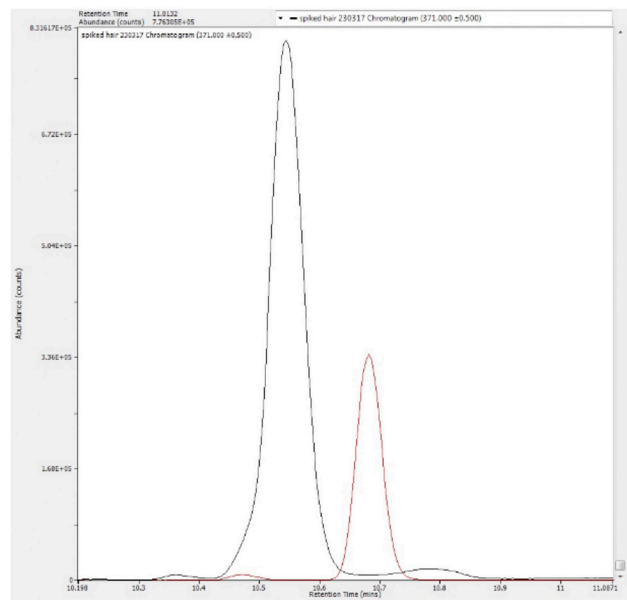


Fig. 5: Comparison of the manual and automated workflow results

Results and Conclusions

The performance of the automated sample preparation was tested with hair samples spiked with known concentrations of traceable standards of THC metabolites. After the derivatization step shown in figure 4, the results from samples prepared using the manual and automated workflows were compared. The results for one of these target compounds are compared in figure 5.

A video of the automated sample preparation workflow can be found here:

https://youtu.be/pkwQmww_4SU

Figure 5 shows that significantly better recovery was achieved by automated sample preparation when compared to manual sample preparation of hair samples spiked at the same concentration.

- Fully automated determination of drugs of abuse in hair samples has been demonstrated, combining automated sample preparation with online injection into any analytical instrumentation
- Multiple extraction techniques can be automatically applied to one digested hair sample and all extracted target compounds combined into one injection for analysis
- Automation provides higher throughput, lower cost, increased reproducibility and can also provide increased sensitivity



<http://www.go-jsb.com>

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