Microsaic Systems

Direct analysis of CBD in Cannabinoid Oils using TLC-MS

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INTRODUCTION

The use of cannabis and cannabis derivatives in medicine and nutritional supplements has now grown internationally, largely due to favourable regulatory status for medicinal use. As more new compounds are released onto the market, accessible techniques are required to quickly and efficiently analyse product quality. In the UK Cannabidiol (CBD) has become popular as a health supplement and many studies have reported analgesic anti-depressant benefits. In its natural form, CBD is derived from the *Cannabaceae* genus of flowering hemp, and the essential oils that form the body of the sample matrix are released during the extraction process. This complex matrix provides a challenge when looking to perform product quality testing. Our application suggests how the QA scientist may extract CBD from the starting matrix, using a hyphenated technique of CAMAG's planar chromatography technology and Microsaic's "point-of-need" mass spectrometer and fluidics handling platform.

EXPERIEMNTAL

CBD samples were obtained in three forms: 2.75% hemp oil and 5% hemp oil droplets and 10mg capsules. These were mixed with methanol and injected into HPLC vials. These were placed into the CAMAG Automatic TLC sampler 4. Samples were applied in narrow bands onto HPTLC plates (HPTLC Si RP-18 F254, 20x10). Vision CATS software was used to create Plates were developed using the CAMAG Automatic Developing Chamber 2. A developing solvent mixture of Methanol: water: acetic acid (70:15:15) was used and then presented using the CAMAG TLC Visualizer 2. Bands were extracted from the developed plate using the CAMAG TLC 2 Interface. The extraction solvent used (Methanol: water ; 80:20 @ 0.5 ml/min) was provided by the Microsaic MiDas™ with the extracted sample passing straight into the Microsaic 4500 MiD•.

Figure 1. System configuration showing the TLC2 interface connected to the 4500 MiD•

RESULTS AND DISCUSSIONS

Developed plates are presented in figure 2. for reference a CBD standard is presented for comparison. Good agreement is seen with the developed band around 0.3 rf giving confidence that the separation has been successful. Mass detection is preferred because direct confirmation is obtained and will also eliminate the need for expensive and tough to acquire reference standards. Information rich data containing co-eluting compounds and matrix impurities are obtained.

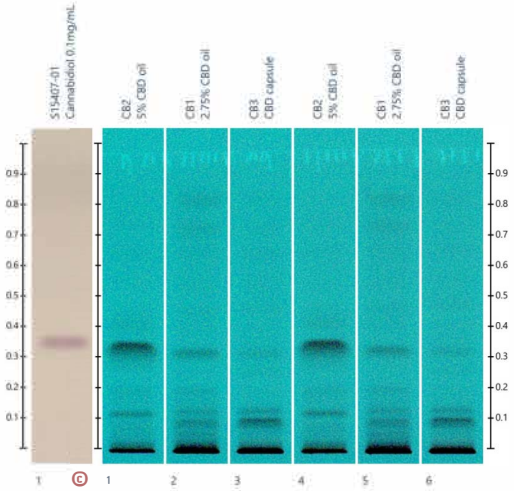

Figure 2. Developed plate showing separated samples. Band at 0.3 rf corresponds well with reported rf value for CBD

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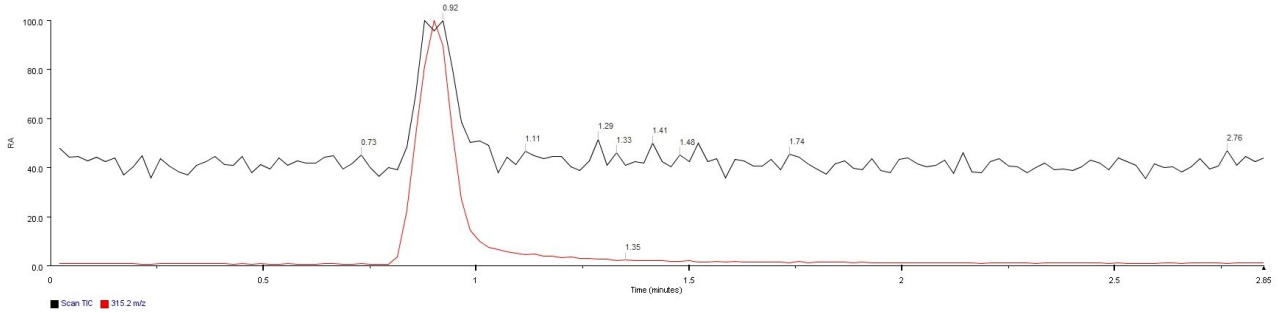

Figure 3. MaSScope data showing combination of TIC over 50-1400u mass range and SIM scan @ 315.2. Bottom shows the mass spectrum that is extracted from the TIC *CBD peak (m/z 315.2; [M+H]+)

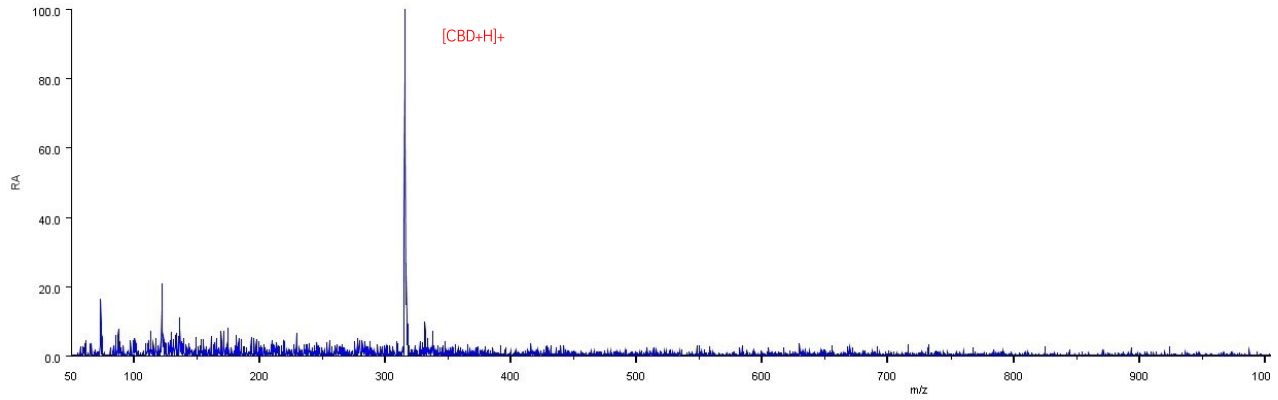

Figure 4. Mass spectrum of extracted band @ 0.3 rf CBD 315.2 m/z; [CBD+H]+

The developed plate was transferred to the TLC Interface 2. The targeting laser was aligned on to the band at 0.3 rf. And the sample extracted, passed through into the solvent stream and through an inline filter to the MiD mass detector.

Figure 3 shows the mass spec data from the developed plate . Good response is seen from a full scan total ion count (TIC) over the full mass range (50-1400 m/z) in tandem with a SIM acquisition that is run in parallel (MaSScope; 315.2 m/z). The signal in the mass spectrum corresponding to [CBD+H]+ is clearly visible in figure 4. This demonstrates the sensitivity of the MiD, and moreover, the flexibility to run two methods in parallel.

CONCLUSIONS

A combination of CAMAG planar chromatography equipment and Microsaic point-of-need technology has been used to extract mass data from popular high-street health supplements. The accessibility of the MiD couples extremely well CAMAG planar technology. The inherent usability means that setup to analysis time is minimised. This means mass analysis of developed plates can occur in line with established work flows.

As Cannabis becomes more accepted in medicine and nutritional supplements, versatile methods are required to meet these challenges. Microsaic Systems Plc and Omicron Research Ltd are committed to bringing these solutions to the point-of-need.

ACKNOWLEDGEMENTS

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