232 North Plaza Drive
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Investigation of the composition of a Δ^8-THC-acetate containing sample SonEx Labs Acetate 656 (SA-06022021-2018)

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Introduction

Sample SonEx Labs Acetate 656 (SA-06022021-2018) was claimed to contain Δ^8-THC-acetate. The sample was analyzed to verify this claim. Although a reference standard for Δ^9-THC-acetate is commercially available, to the best of our knowledge, a reference standard for Δ^8-THC-acetate is not. Therefore, we could not verify the identity of the submitted material as Δ^8-THC-acetate by comparison to a certified reference standard. However, the sample was analyzed by GC/MS and HPLC_DAD, and the resulting data were interpreted and compared to published data for Δ^8-THC-acetate and laboratory data for Δ^9-THC-acetate.

Note that Δ^9-THC acetate and Δ^9-THC-O-Acetate are used interchangeably to refer to the same substance.

Experimental

The following samples were prepared:

Δ^9-THC-acetate solution:

- In a vial, 10 µL of Δ^9-THC-O-Acetate solution (Cerilliant T151-1ML, lot FE03192002, 1 mg/ml) was pipetted.
- The solution was diluted with 990 µL of acetonitrile.
- The final concentration of Δ^9-THC-acetate was 10 µg/mL.

Sample SA-06022021-2018 solution:

- In a 20-mL scintillation vial, 82.12 mg of sample SA-06022021-2018 was weighed out.
- The sample was dissolved with 8.2 mL of acetonitrile.
- The sample concentration of the intermediate solution 1 was 10 mg/mL.
- Into a vial, 10 µL of intermediate solution 1 was pipetted.
- To the vial, 990 µL of acetonitrile was added.
- The sample concentration of the intermediate solution 2 was 100 µg/mL.
- Into a vial, 100 µL of intermediate solution 2 was pipetted.
- To the vial, 900 µL of acetonitrile was added.
- The final sample concentration was 10 µg/mL.

Mixed solution:

- Into a vial, 100 µL of Δ^9-THC-acetate solution was pipetted.
- To the vial, 100 µL of sample solution was pipetted.
- The final concentrations were 5 µg/mL of Δ^9-THC-acetate and 5 µg/mL of sample SA-06022021-2018 or Δ^8-THC-acetate (assuming sample SA-06022021-2018 is pure Δ^8-THC-acetate).

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All samples were analyzed by GC-MS. Two events were set-up:

1. A full MS1 mass scan with a scan range of m/z 45–450 to record mass spectral data that is used for identification of the compounds.
2. A SIM containing m/z 356 (monoisotopic mass of Δ^8- and Δ^9-THC-acetate) and m/z 314 (monoisotopic mass of Δ^8- and Δ^9-THC) to record the signal of the analytes Δ^8- and Δ^9-THC-acetate and any presence of Δ^8- and Δ^9-THC.

Sample SA-06022021-2018 solution was also analyzed by HPLC-PDA.

Results and discussion

GC-MS results

In the full-scan TIC (total ion chromatogram) of the Δ^9-THC-acetate solution (Fig. 1), a signal was present at 6.07 min. The mass spectrum at this retention time (Fig. 2) confirmed the presence of Δ^9-THC-acetate through a library match (Fig 3).

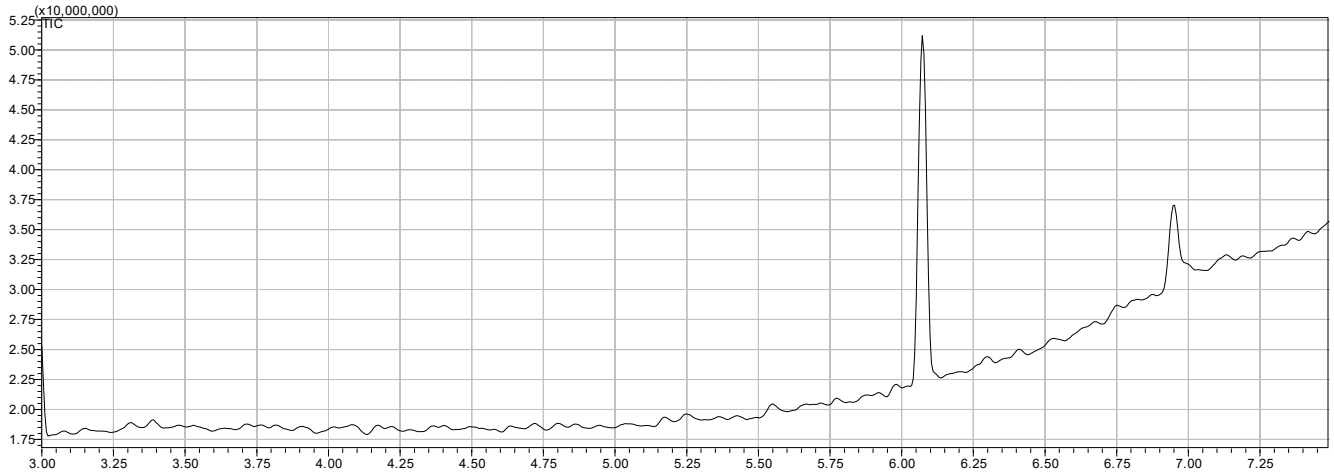


Figure 1: TIC of the full-scan of the Δ^9-THC-acetate solution.

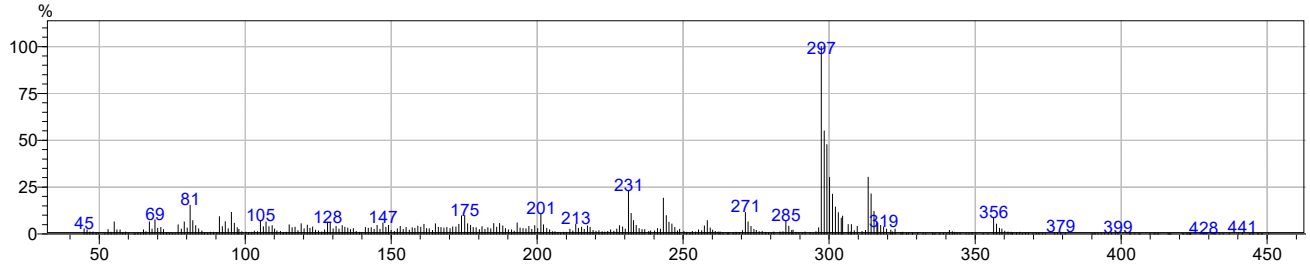


Figure 2: Mass spectrum of at retention time 6.07 min of the Δ^9-THC-acetate solution.

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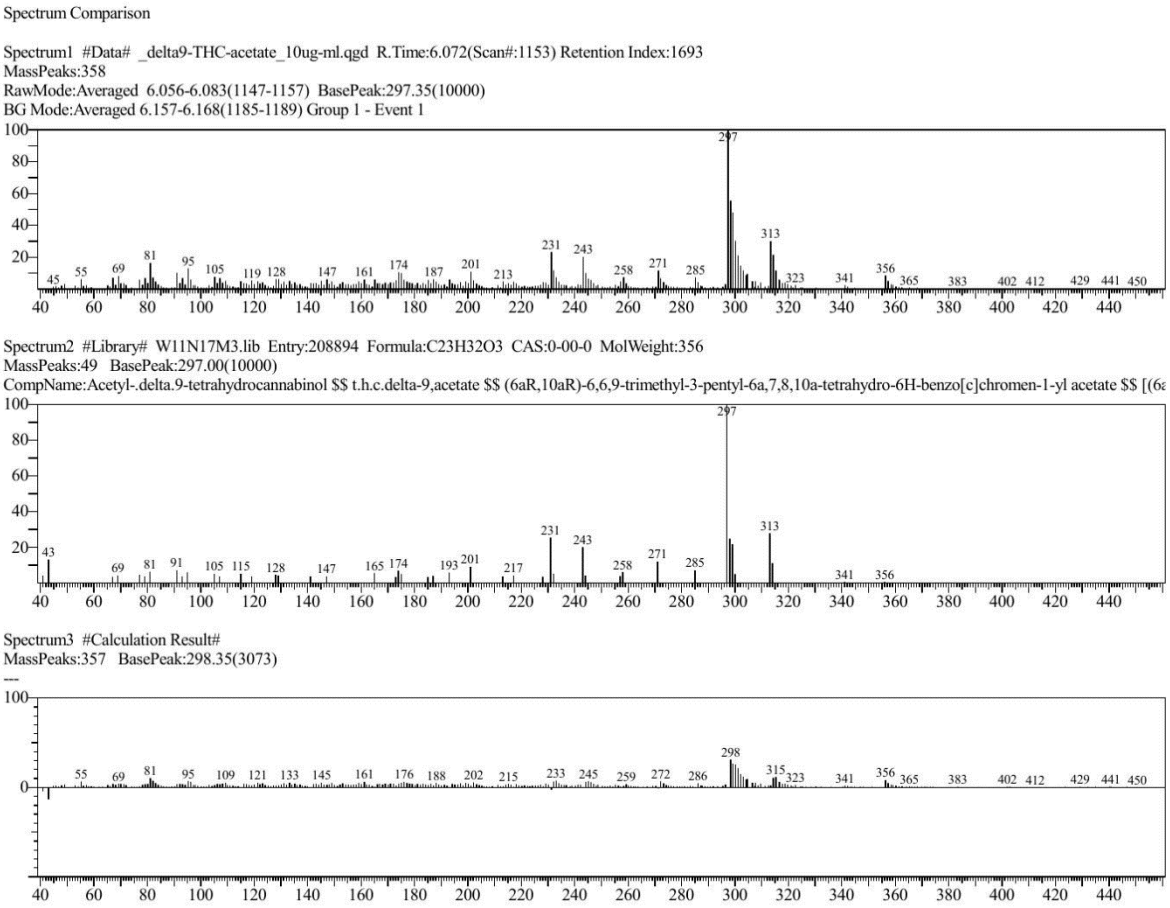


Figure 3: Library match of the mass spectrum at retention time 6.07 min of the Δ^9-THC-acetate solution.

In the full-scan chromatogram of sample SA-06022021-2018 (Fig. 4), a signal was present at 5.98 min. The mass spectrum at this retention time (Fig. 5) confirmed that this compound is Δ^8-THC-acetate through a library match (Fig 6).

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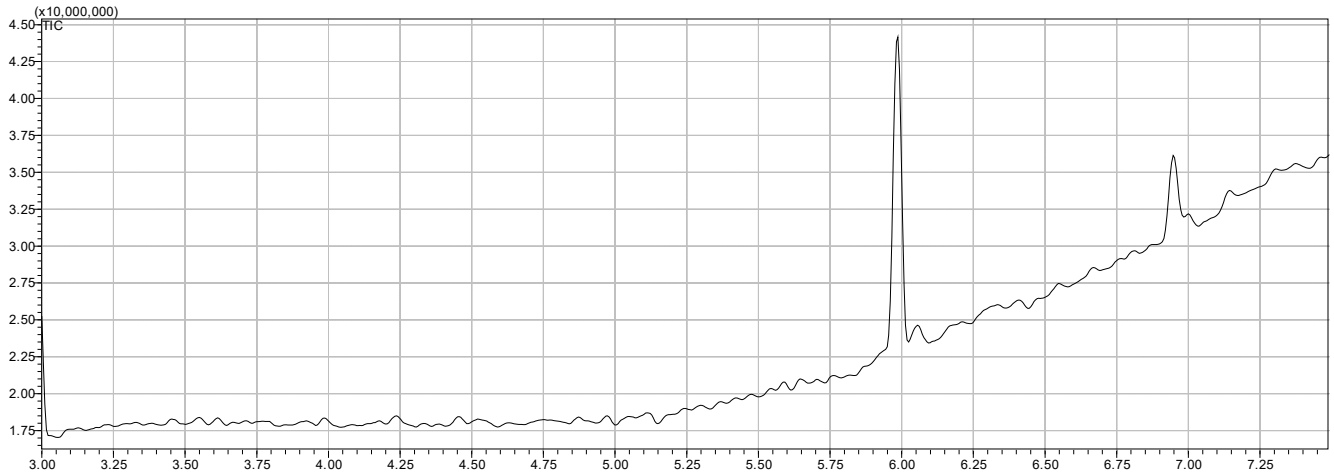


Figure 4: TIC of the full-scan of the sample SA-06022021-2018 solution.

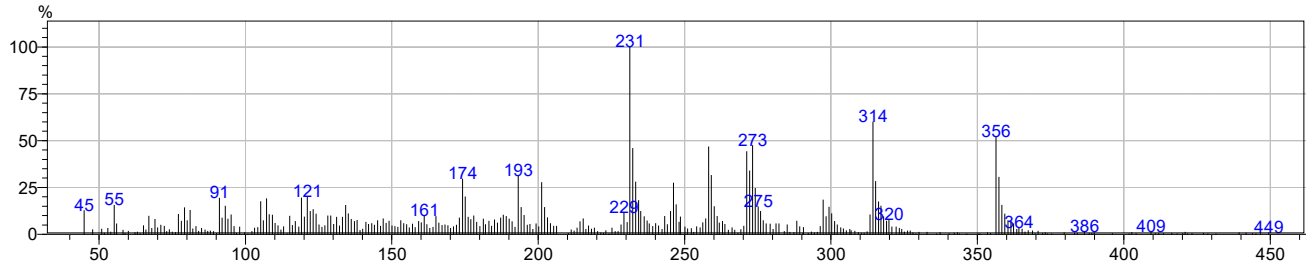


Figure 5: Mass spectrum of at retention time 5.98 min of the sample SA-06022021-2018 solution.

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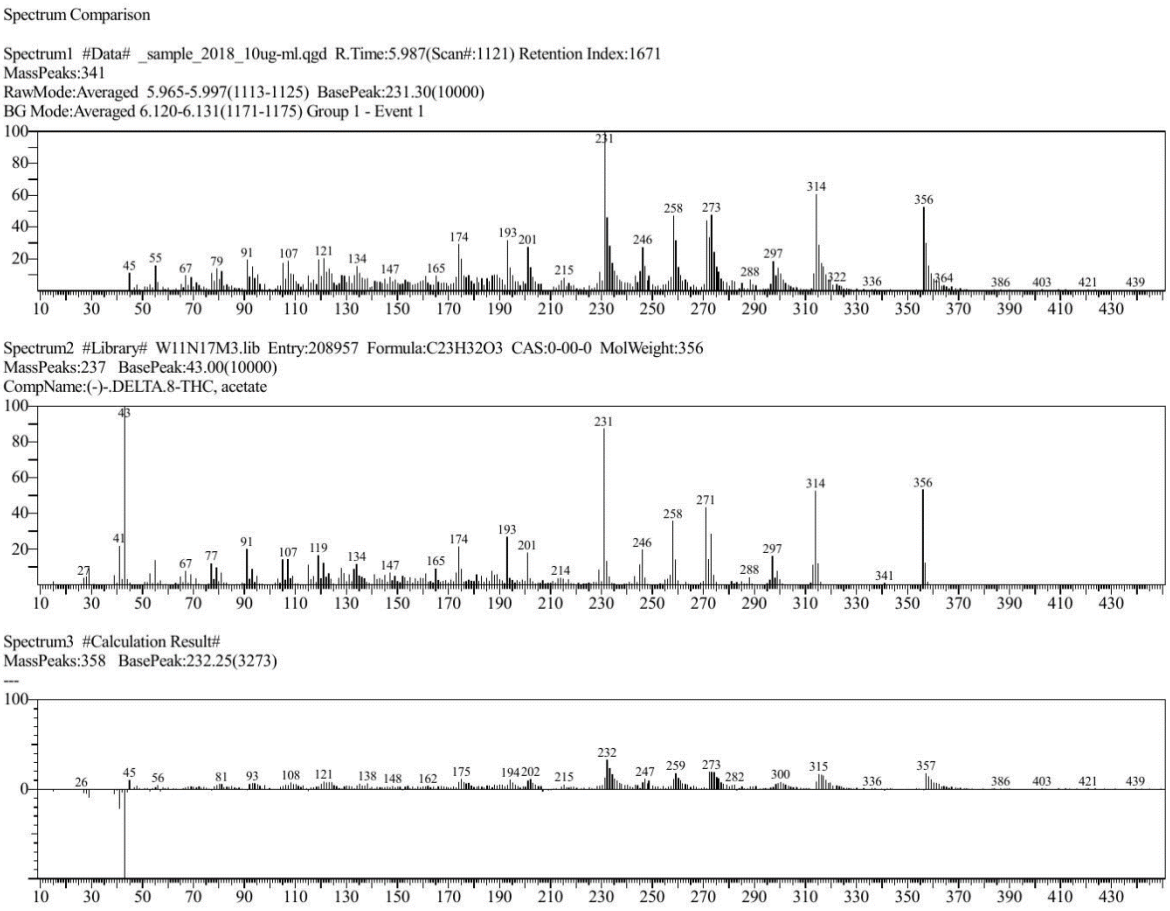


Figure 6: Library match of the mass spectrum at retention time 5.98 min of the sample SA-06022021-2018 solution.

The extracted ion chromatogram of m/z 365 of the mixed solution (Fig. 7) indicated that Δ^8- and Δ^9-THC-acetate were chromatographically resolved. The order of elution of the acetate esters is the same as that for Δ^8-THC and Δ^9-THC.

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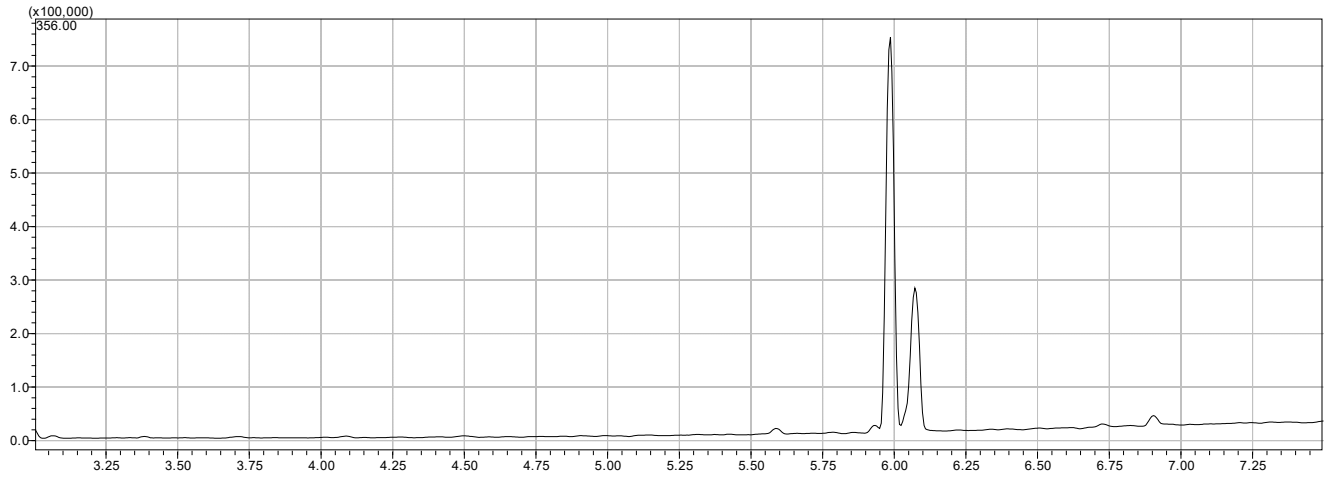


Figure 7: SIM chromatogram of m/z 365 of the mixed solution. The signals at retention times 5.9 and 6.1 min were attributed to Δ^8- and Δ^9-THC-acetate.

The certificate of analysis for Δ^9-THC-acetate indicated that only 0.15 % Δ^9-THC and < 0.01 % CBDVA were present in the standard. However, small amounts of Δ^8-THC and Δ^9-THC were detected in the reference standard solution of Δ^9-THC-acetate (Fig. 8). Similar signal intensities for Δ^8-THC were also observed in the solvent blanks that were analyzed. It was therefore concluded that the signal for Δ^8-THC in the analytical results of this sample was probably due to internal contamination of the system from previously analyzed concentrated Δ^8-THC containing samples. Therefore, it could not be determined whether the analyte was present in the sample.

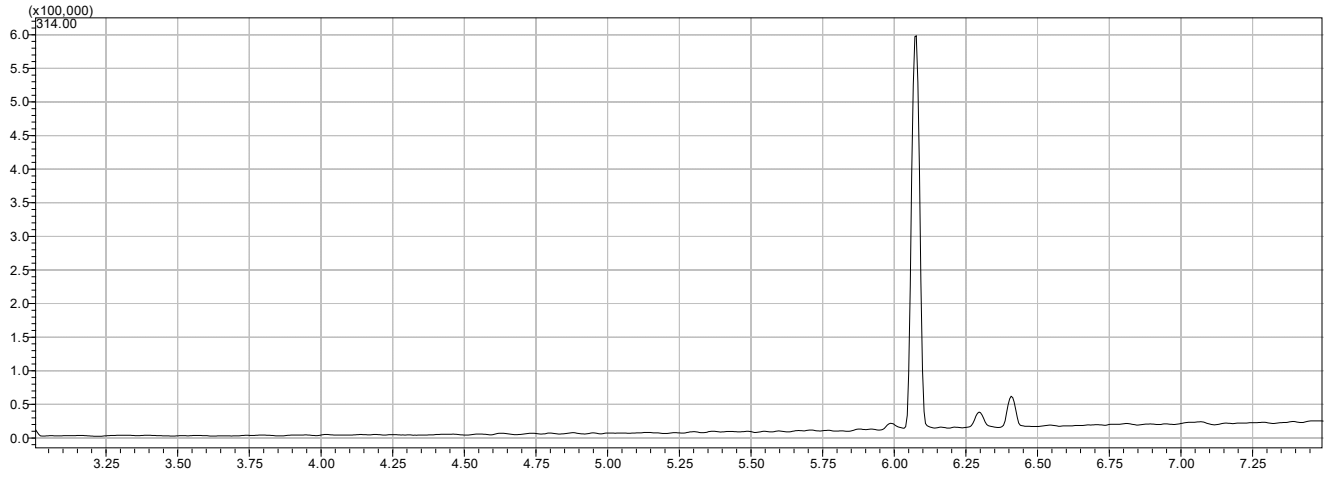


Figure 8: SIM chromatogram of m/z 314 of the Δ^9-THC-acetate solution. The signals at retention times 6.1, 6.3 and 6.4 min were attributed to Δ^9-THC-acetate, Δ^8-THC, and Δ^9-THC.

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Sample SA-06022021-2018 contained mainly Δ^8-THC-acetate (Fig. 9). However, small amounts of Δ^9-THC-acetate were detected by as well. As previously stated, the signal of Δ^8-THC was possibly due to an internal contamination of the system with this compound.

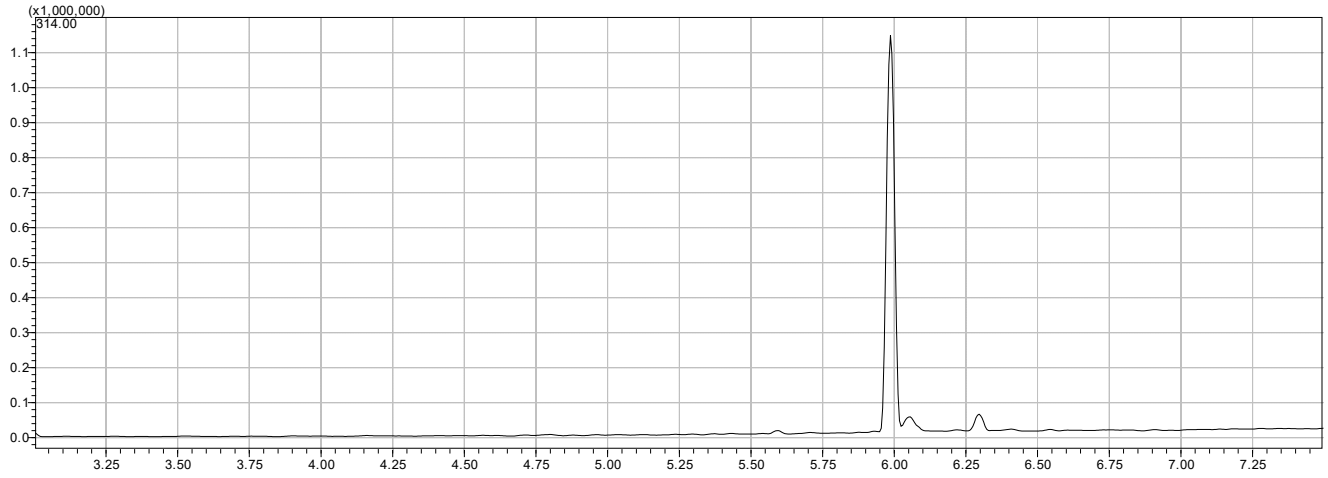


Figure 9: SIM chromatogram of m/z 314 of the sample solution. The signals at retention times 5.9, 6.1 and 6.3 min were attributed to Δ^8-THC-acetate, Δ^9-THC-acetate, and Δ^8-THC.

HPLC-PDA results

From the HPLC results of sample SA-06022021-2018, it was estimated that the relative area contribution of Δ^8-THC-acetate was 80 % of the total peak area. The relative area contributions of Δ^8- and Δ^9-THC were 2.2 % and 0.24 %. An peak in the HPLC chromatogram had a relative area contribution of 9.2 % of the total.

Note that all these numbers were based on estimates. Furthermore, the attributed signal for Δ^8-THC-acetate was cut off because the run time was too short since the sample was analyzed using the standard cannabinoids method. For future analyses, the run time will need to be extended to get more accurate values.

Conclusions

GC-MS analysis and spectral library matching indicated that sample identified as SonEx Labs Acetate 656 (SA-06022021-2018) contains Δ^8-THC-acetate and possibly a small amount of Δ^9-THC-acetate. Minor amounts of Δ^8- THC and Δ^9-THC were detected in the sample but the Δ^8- THC was almost certainly a contaminant.

Due to the unavailability of a certified reference standard of Δ^8-THC-acetate, it was not possible to quantify the amount of Δ^8-THC-acetate in the sample. However, the area of the peak attributed to Δ^8-THC-acetate in the HPLC-PDA analysis of the sample represented at least 80% of the total area of all peaks detected.