Application Note

GC Separation of ADB-BUTINACA from its Precursor ADB-INACA

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Key Features

- Coelution of ADB-BUTINACA and its synthetic precursor ADB-INACA makes traditional GC-MS methods difficult for the quantification of these two novel psychoactive substances (NPS). Quantities of these synthetic cannabinoids are distorted if the testing methods are unable to resolve the two substances in analyzed samples.
- A new GC method for the separation of ADB-BUTINACA from its precursor ADB-INACA offers a reliable and robust solution for identifying this DEA Schedule I compound.

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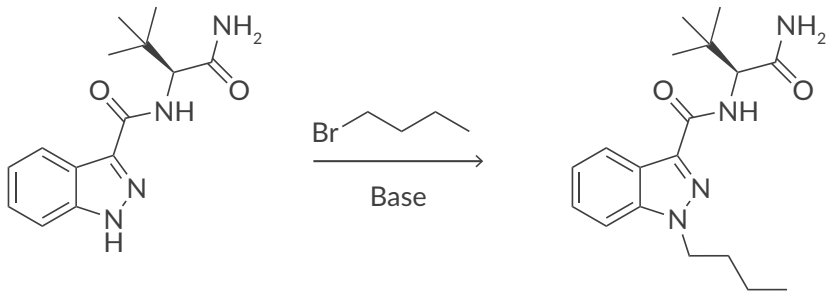
Introduction

Following China's structural class ban on synthetic cannabinoids in 2021, the novel psychoactive substance (NPS) structural landscape has been constantly evolving to evade these regulations. While new structural variations may appear on the grey market, a new phenomenon is the emergence of DIY semi-finished kits. These semi-finished kits provide the end-user with non-regulated precursors enabling the user to finish the synthesis themselves to the desired synthetic cannabinoid of choice.[1] Several of these tail-less precursors have been detected on the grey market since late 2021. One such tail-less precursor is ADB-INACA, which was first detected in the United States in November of 2022.[2,3] ADB-INACA may be a penultimate synthetic intermediate towards ADB-BUTINACA **(Figure 1)** as well as other synthetic cannabinoids such as 5-fluoro ADB-PINACA and ADB-4en-PINACA.



ADB-INACA ADB-BUTINACA

Figure 1. Final step in the synthesis of ADB-BUTINACA from ADB-INACA.

ADB-BUTINACA (a.k.a. ADB-BINACA), was first detected in Europe in September of 2019 *via* the European Union Drugs Agency (EUDA) and was not detected in the United States until July 2020.[4,5] Positive identifications in seized suspect drug materials continued to increase in the United States in 2021 and as of 2024 have remained constant.[6]

The coelution of ADB-BUTINACA and its precursor poses a challenge for forensic chemists and distorts reporting methods when they cannot be resolved. This application note highlights the coelution observed between ADB-BUTINACA and its precursor ADB-INACA and presents a new and improved GC method that provides baseline separation of the two species.

Methods

Both components at 1 mg/ml and a mixture of the two solutions were analyzed on an Agilent 8890 GC equipped with a 5977B MS detector. The injector temperature was set at 300°C, and the oven temperature was programmed to start at 50°C, hold for 1 minute, and then increase at a rate of 30°C per minute up to 300°C. The total run time was 15 minutes. The injection mode was set to a 15:1 split. Helium was used as the carrier gas at a constant flow rate of 2 ml/min. The single component of the ADB-BUTINACA and ADB-INACA solution was used to confirm the retention time order in the mixture.

A mixture consisting of equal parts ADB-BUTINACA and ADB-INACA was initially injected on a DB-5MS column, a non-polar GC column equivalent to (5%-phenyl)-methylpolysiloxane (USP phase G27), which resulted in significant coelution of ADB-BUTINACA and ADB-INACA **(Figure 2)**.

Analyte	Retention Time (min)	Cayman Item No.
ADB-BUTINACA	11.849	29350
ADB-INACA	11.876	37729

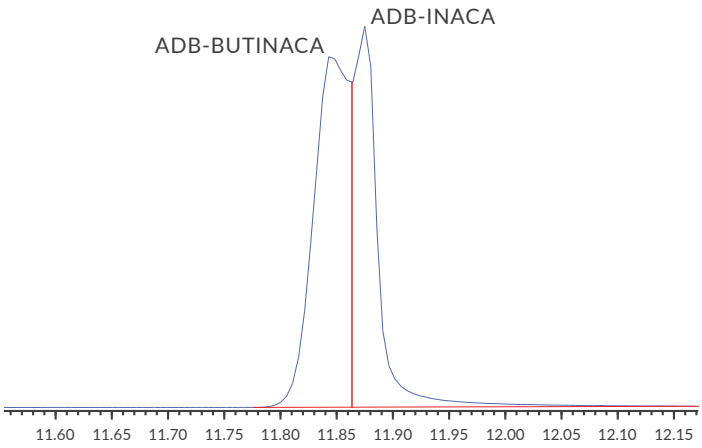


Figure 2. Co-injection of ADB-INACA and ADB-BUTINACA on a DB-5MS column.

Analyte	Retention Time (min)	Cayman Item No.
ADB-BUTINACA	11.370	29350
ADB-INACA	11.886	37729

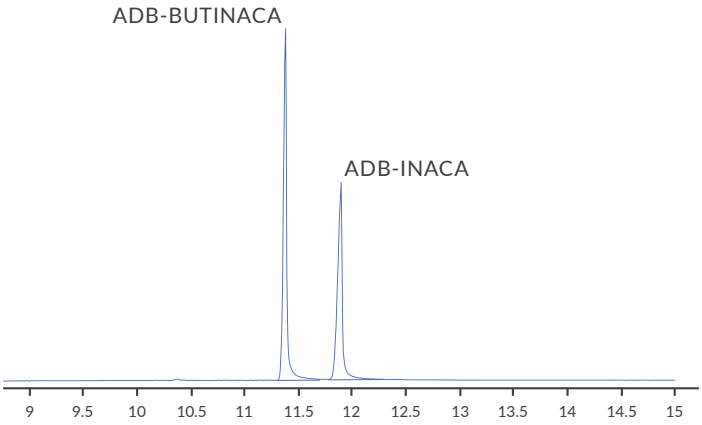


Figure 3. Co-injection of ADB-INACA and ADB-BUTINACA on a DB-35MS column.

When attempts to resolve the peaks by adjusting the oven temperature were not successful, the solution mixture was then introduced on to a DB-35MS column, which is equivalent to a (35%-phenyl)-methylpolysiloxane (USP phase G42) with mid-polarity. The results with this column showed that all peaks were now well separated, with a peak resolution greater than 1.5 (**Figure 3**). This provides excellent EI-MS spectra to clearly differentiate between ADB-BUTINACA and ADB-INACA, as shown in **Figures 4 and 5** (proposed fragmentation included).

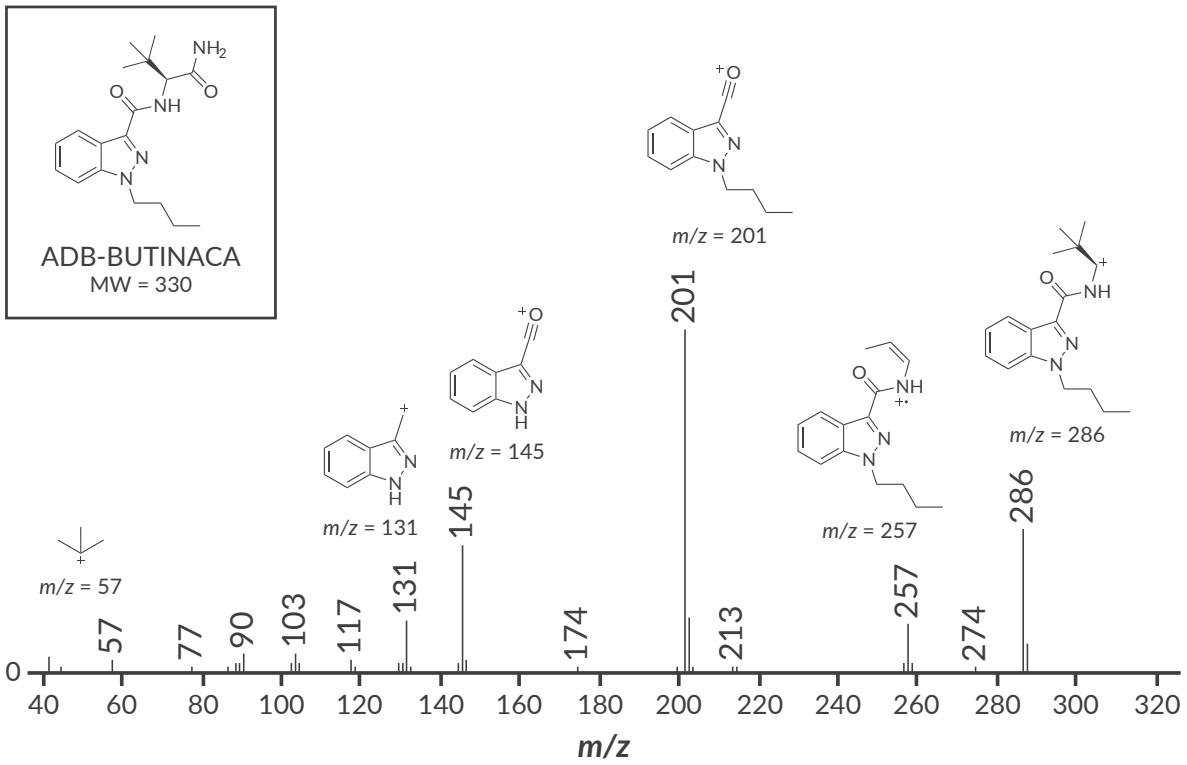


Figure 4. EI-MS spectrum of ADB-BUTINACA.

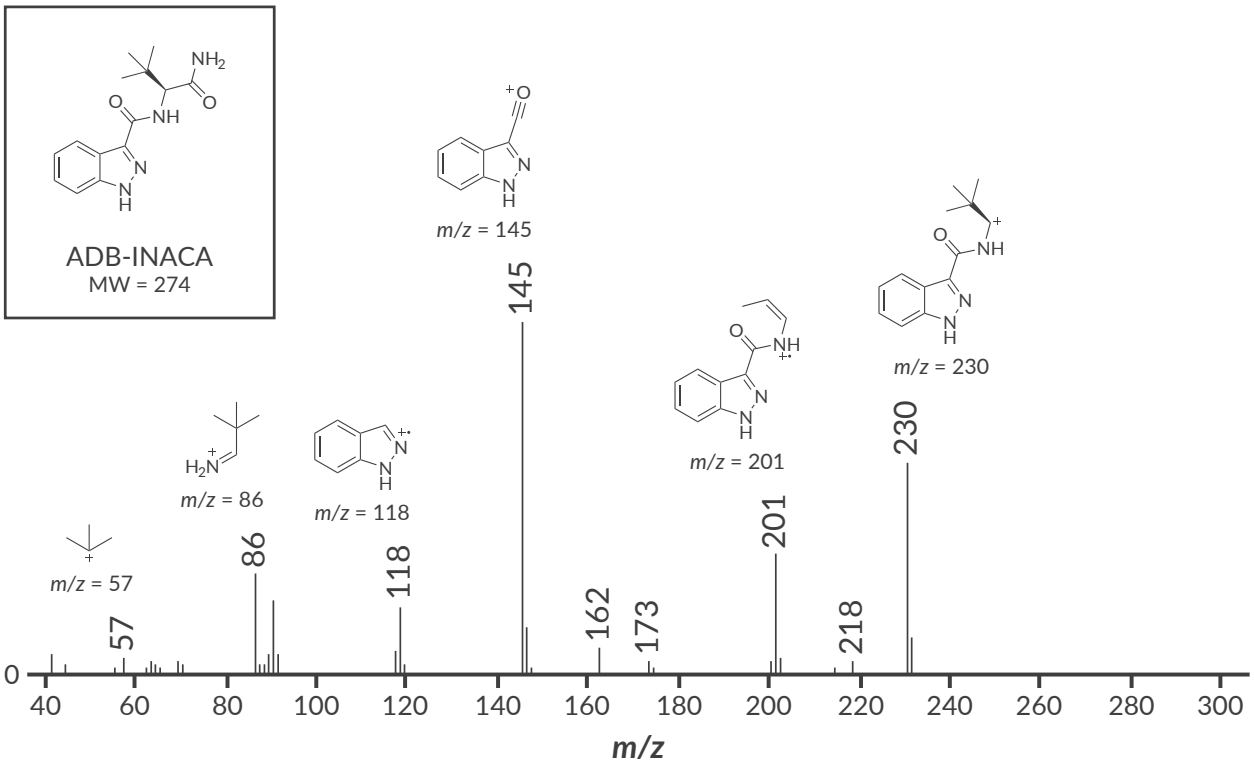


Figure 5. EI-MS spectrum of ADB-INACA.

Conclusions

The coelution of ADB-BUTINACA and its precursor ADB-INACA on traditional GC-MS columns poses a challenge because the mass spectrum of the combined compounds may lead to the inability to positively identify the presence of a DEA Schedule I substance and lead to the misinterpretation of the data as a new unknown substance. This application note has successfully differentiated ADB-BUTINACA from its precursor ADB-INACA by using GC-MS equipped with a DB-35MS column (or equivalent column). The information provided here can help identify and differentiate both products as well as other analogs in forensic casework. The GC separation conditions can also be used to quantify ADB-BUTINACA and ADB-INACA.

References

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