Rapid Characterization of Seized Street Drugs using Evolved Gas Analysis plus Multi-Step Pyrolysis

Application Note
Forensic Science

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Abstract
This application note demonstrates a fast Py-GC-MS testing method for seized street drugs

Introduction

Common screening methods for drugs of abuse from biological samples are immunoassays, which usually performs by GC-MS or LC-MS. In the sample preparation, the first step is a few hours of enzymatic or acidic hydrolysis prior to the analysis. If the testing method is GC-MS, additional acidic derivatization is required to make the analytes more volatile, which is prone to uncertainties like reagent quality and other interferences. If LC-MS is used, such derivation steps are skipped and could shorten the additional sample preparation steps after hydrolysis from 6 hour/day to 2 hour/day by using commercial kit.

Same testing techniques are generally used in screening seized street drugs, where these extensive sample preparation procedures are performed to remove the matrices to test the active compounds in the drug. The chemical structure information on the binders, fillers, and adulterants are lost from such tests regardless using GC-MS or LC-MS method.

In recent years, trends of illicit drugs are continuously changing throughout all regions of the United States. According to the CDC, in 2015 synthetic opioids, which are mostly fentanyl, surpassed heroin among drug overdose deaths in the U.S. Due to the fact that the elimination half time of fentanyl is only a few hours, the drugs of abuse test from biological sample is challenging and the demand on rapid testing of seized drugs are increasing.

In this application note, Evolved Gas Analysis (EGA) followed by Multi-Step Pyrolysis (MSP) using a Pyroprobe are demonstrated as a quick and simple screening method to analyze a seized street drug sample including adulterants and fillers.

Experiment Setup

Two 100 μg samples of a seized street drug powder were each placed in Drop-In-Sample-Chamber (DISC) tubes for Evolved Gas Analysis (EGA) and Multi-step Pyrolysis (MSP), respectively, on a Pyroprobe 6150 Autosampler.

EGA

Pyroprobe 6150 Autosampler
Initial: 50°C
Final: 1000°C
Ramp Rate: 100°C per min
Interface: 300°C
Transfer Line: 325°C
Valve Oven: 300°C

Thermo Trace 1310 GC-MS
Column: Fused silica (1m x 0.10mm)
Carrier: He1.25mL/min 75:1 spl
Injector: 360°C
Oven: 300°C
Ion Source: 230°C
Mass Range: 35-600amu

Multi-Step Pyrolysis

Pyroprobe 6150 Autosampler

DISC:
200°C 60 sec
250°C 60 sec
300°C 60 sec

Interface: 300°C
Transfer Line: 325°C
Valve Oven: 300°C

Thermo Trace 1310 GC-MS

Column:
5% phenyl (30m x 0.25mm x 0.25μm)
Car: He 1.25mL/min, 75:1 spl
Injector: 360°C
Oven: 40°C for 2 min
12°C/min to 320°C (10min)
Ion Source: 230°C
Mass Range: 35-550amu

Results and Discussion

Figure 1 shows an EGA run of the powder. The bulk of the sample begins degrading at 225°C, peaks at 275°C, and is completely degraded by 450°C.

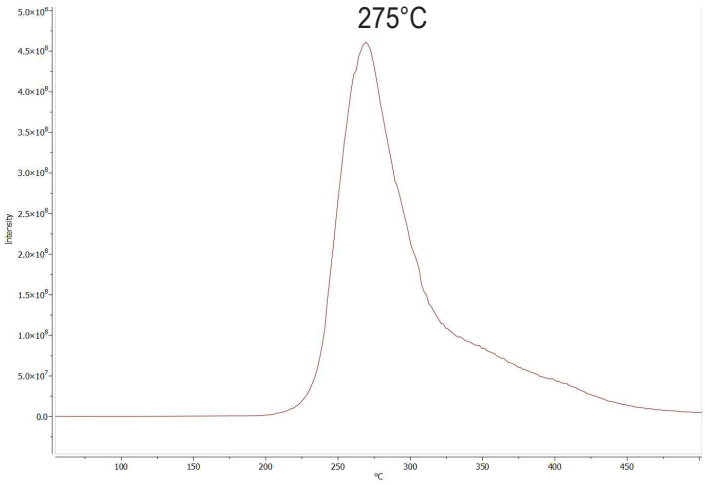

Figure 1. EGA of illicit drug powder.

The powder was further investigated by performing multi-step pyrolysis. As the Pyroprobe 6150 autosampler can perform an unlimited amount of temperature steps, 3 temperatures were chosen, from 200°C to 300°C at 50°C increments, to gain more information about the entire contents of the powder (Figure 2). At the first temperature, 200°C, fentanyl and a direct precursor to fentanyl, 4-anilino-N-phenethylpiperidine (4-ANPP), are present.

At 250°C more fentanyl is seen, and degredation peaks of the main filler also emerge. After the automatic integration of all the peaks and manually clearing peaks already identified as fentanyl and 4-ANPP, the mass spectra of remaining peaks were co-added together by using the Peak(background subtr.) tool in MSChrom. The resulting mass spectra searched against the pyrolysis database had a top match for cellulose, which is a common drug filler.

By 300°C , much the filler has been removed, and only fentanyl remains. It appears that despite fentanyl's high boiling point, over 400°C[1], vaporization can occur at a much lower temperature.

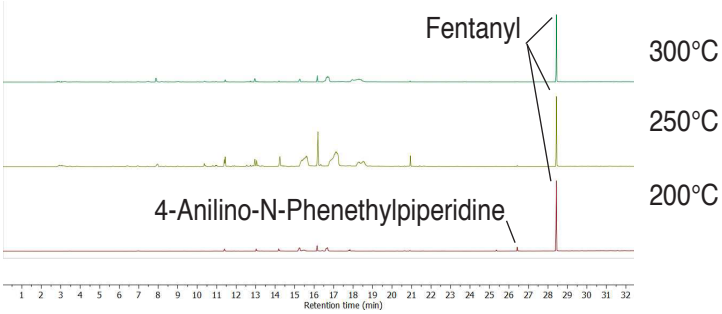

Figure 2. Multi-step pyrolysis of drug powder, from 200°C (bottom), to 300°C(top).

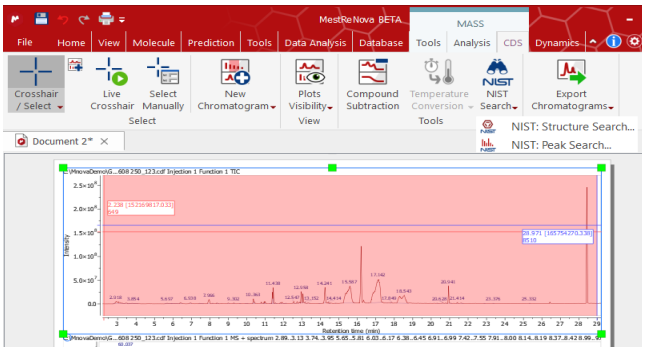

Figure 3. Co-adding integrated peaks of 250°C run to perform a pyrolysis library search.

The EGA + MSP technique provides quick sample prep, creating a quick and easy screening technique to analyze the drug in its entirety, which includes active compounds, adulterants and fillers.

Conclusion

EGA and MSP are two powerful tools in material investigation, including illicit drugs. The first tool can quickly screen the sample based on temperature, whereas the second tool will give detailed insight on the chemical composition. From this investigation, we can see that fentanyl can vaporize from a drug sample at a low temperature, 200°C. Also, a drug pre-cursor, as well as the main filler was identified. This is but one illustration of the possible advantages of integrating multi-step pyrolysis with EGA, which have the potential of replacing time-consuming analytical methods.

References

1. EPA Fact Sheet for OSCs: Fentanyl and Fentanyl Analogs Version 1.0 05/22/2018.