Application Note: 432

Identification of Lysergic Acid Diethylamide (LSD) in Candy by UHPLC/MS

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Key Words

- Accela™ UHPLC
- MSQ Plus™
- Hypersil GOLD™ PFP
- Forensic Analysis
- LSD

Goal

Positively identify trace amounts of lysergic acid diethylamide (LSD) in sugar candy quickly, with minimal sample preparation and no chemical derivatization.

Introduction

Lysergic acid diethylamide (LSD) is a controlled substance in forensic chemistry that is notorious for being difficult to identify. Its myriad evidentiary forms include paper tabs, eye drops, sugar cubes and small sugary candies such as sweet tarts, valentine hearts or mints. Because it is such a potent hallucinogen, typical street doses require only 40 to 120 µg of LSD. The small personal-use amounts seized by state and local law enforcement often lack sufficient drug to allow both forensic analysis by traditional means and archiving of some of the evidence for follow-up testing.

Most forensic laboratories confirm the presence of LSD by using gas chromatography with mass spectrometry (GC/MS). LSD is extracted from the evidence with an organic solvent, derivatized, and determined by GC/MS. GC/MS resolves LSD from other compounds and provides structural information that can be compared to reference spectra in a searchable library.

The disadvantages of GC/MS are its requirements for extensive sample preparation, including chemical derivatization of LSD to a more volatile form, and its impaired performance with analytes that are polar, thermally labile, or nonvolatile. LSD has a high affinity for active sites in liners that can spoil chromatographic resolution. LSD-doped sugar cubes or candy can foul the GC with sugars, increasing the burden of instrument maintenance and hindering throughput.

An alternative method to positively identify LSD in complex food matrices is to use ultra high performance liquid chromatography with mass spectrometric detection (UHPLC/MS). UHPLC/MS offers a threefold benefit compared to GC/MS; simpler sample preparation, no derivatization, and less time wasted baking out or cleaning the instrument. This application note demonstrates how a working forensic laboratory uses UHPLC/MS to analyze sugar candies for LSD. LSD in doped sugar cubes and candy hearts is simply extracted, separated within 5 minutes on a Hypersil GOLD PFP 1.9 µm column, and confirmed by a fast scanning single quadrupole mass spectrometer.

Experimental Conditions

1. Standard and Sample Preparation

A 1000 mg/L solution of LSD in methanol was purchased from Alltech (State College, PA, USA) and diluted to about 5 mg/L with methanol.

The sugar cubes and candy hearts were purchased from a local grocery store. The candy hearts and sugar cubes were treated with 3-5 drops of this LSD solution and allowed to stand for 24 hours prior to use. Ten (10) mg scrapings from the sugar cube or candy heart were added to 2 mL methanol. This mixture was vortexed for 30 sec, allowed to settle for 1 min, and the supernatant was filtered through a cotton-plugged Pasteur pipette. The filtrate was centrifuged for 90 sec, and the supernatant was filtered through a second cotton-plugged Pasteur pipette and transferred to the autosampler vial.

2. Chromatographic Conditions

Chromatographic analyses were performed using the Accela UHPLC system (Thermo Fisher Scientific, San Jose, CA). The chromatographic conditions were as follows:

Column:	Hypersil GOLD PFP (perfluorinated phenyl) 1.9 µm, 100 x 2.1 mm			
Flow Rate:	1 mL/min			
Mobile Phase:	A: Water with 0.06 % acetic acid B: Acetonitrile (ACN) with 0.06% acetic acid C: Methanol with 0.06% acetic acid			
Gradient:	T (min)	A%	B%	C%
	0.00	95.0	0.0	5.0
	1.00	95.0	0.0	5.0
	1.50	90.0	5.0	5.0
	2.70	70.0	10.0	20.0
	3.00	5.0	15.0	80.0
	7.00	5.0	0.0	95.0
	7.10	95.0	0.0	5.0
	8.00	95.0	0.0	5.0
Column Temperature:	45 °C			
Injection:	2 µL partial loop injection, 25 µL loop size Syringe Speed: 8 µL/sec Flush Speed: 100 µL/sec Flush Volume: 400 µL Wash Volume: 100 µL Flush/Wash source: Bottle with methanol			

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3. Mass Spectrometer Conditions

MS analysis was carried out on a MSQ Plus single quadrupole LC/MS detector (Thermo Fisher Scientific, San Jose, CA). The MS conditions were as follows:

Ionization:	Electrospray (ESI)
Polarity:	Positive
Probe Temperature:	500 °C
Cone Voltage:	90 V
Scan Mode:	Full scan with mass range of *m/z* = 125-425 amu
ESI Voltage:	3.5 kV
Scan Time:	0.2 s

Results

LSD elutes at 4.49 min and is detected by using full scans (*m/z* = 125 - 425) of the single quadrupole mass spectrometer. The extracted ion chromatograms from *m/z* 324 ± 0.5 are displayed in Figure 1A. The MS spectrum of the LSD standard shows two molecular ion signals: $[M+H]^+$ at *m/z* 324.1, and $[M+ACN+Na]^+$ at *m/z* 387.1. The MS spectrum of LSD also shows two fragment ion signals at *m/z* 223.3 and 281.3 (Figure 2A).

The methanol extracts from the candy hearts and sugar cubes, doped with LSD, were analyzed using the same UHPLC/MS method as for the standard LSD (Figure 1B, 1C). Positive confirmation of LSD in the samples is assured both by retention time matching and MS spectra matching of the samples (Figure 2B-C) with the LSD standard.

Conclusion

UHPLC/MS can positively identify trace amounts of lysergic acid diethylamide (LSD) in sugar candy in 8 min, after a simple 10 min sample prep involving no chemical derivatization.

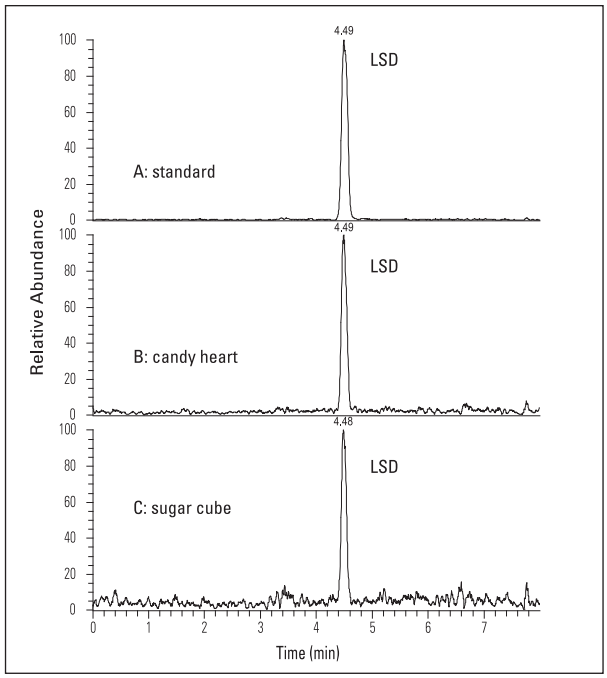

Figure 1: Extracted ion chromatograms at *m/z* = 324 ± 0.5 amu obtained by UHPLC/MS on a Hypersil GOLD PFP column: (A) LSD standard; (B) methanol extract of LSD-doped candy heart; (C) methanol extract of LSD-doped sugar cube. See text for details.

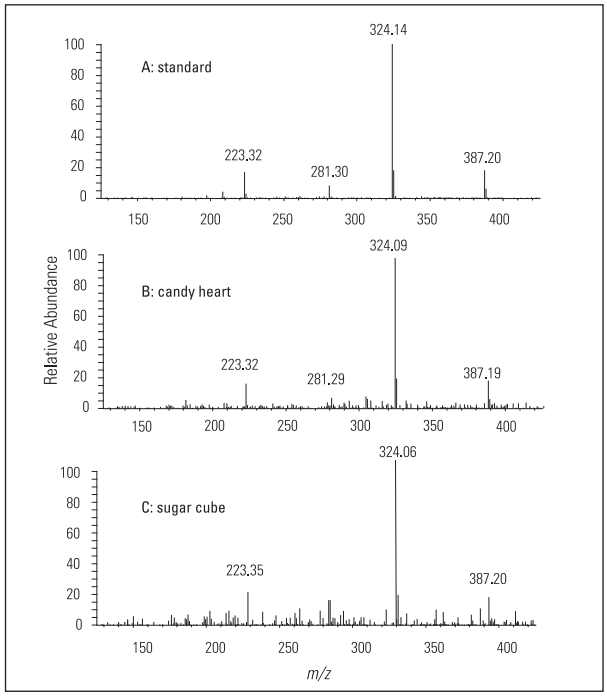

Figure 2: MS spectra of LSD obtained by UHPLC/MS on a Hypersil GOLD PFP column: (A) LSD standard; (B) methanol extract of LSD-doped candy heart; (C) methanol extract of LSD-doped sugar cube. See text for details.

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