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Lysergic Acid Diethylamide Analytical Method

1.0.0 Background

LSD was originally synthesized from lysergic acid found in the fungus *claviceps purpurea*. Street LSD is found most often on blotter paper. It is also found on sugar cubes, candies like “Sweet Tarts”, gelatin squares called windowpanes, and on small pills called microdots. It breaks down in the presence of light and heat, because of this the samples are often found wrapped in metal foil.

2.0.0 Scope

The following analytical procedures are used to confirm the presence of lysergic acid diethylamide (LSD).

3.0.0 Equipment and Reagents

The following pieces of equipment can be used to identify the analyte of interest.

- 3.1.0 A GC/MS and appropriate analytical software. Reference GC/MS AM.
- 3.2.0 Ultraviolet light source.
- 3.3.0 Thin Layer Chromatography (TLC) plates and tank.
- 3.4.0 ACS grade solvents.
- 3.5.0 Distilled or deionized water.
- 3.6.0 NaHCO₃ or Na₂CO₃

4.0.0 Ultraviolet (UV) Test

Although by no means definitive, this test can be used as a presumptive test. Place the evidence under the UV. The suspected LSD should glow a light violet-blue. This test is especially useful in identifying which side of a sugar cube, or candy, has been spiked with LSD. It is common for white paper to reflect the UV and appear violet even without LSD.

5.0.0 GC/MS Sample Preparation and Analysis

- 5.1.0 Sample preparation. As with all GC analyses it may be necessary to concentrate the extracts from either of the following methods; this is done by blowing a stream of air, or other suitable gas, over the top of the solvent. Do not heat!
 - 5.1.1 “Window panes”, blotter paper, and pulverized microdots can be extracted directly with methanol. Place sample in a test tube and add just enough methanol to cover sample. Shake and then let soak for at least an hour. Microdots should soak overnight if possible. Centrifuge if necessary and analyze.
 - 5.1.2 Sugar cubes, “Sweet Tarts” or other candy. Check under UV to find the side that is suspected of being spiked. Scrape off upper layer until

approximately one half of the sample, has been used. Dissolve in water and make basic. Extract with chloroform. Analyze on GC/MS. Using the extraction procedure in 5.2.2, without the derivatizing agent, also works well.

5.1.3 Due to the typically dilute nature of LSD samples, the GC should be set to splitless mode. The injector liner may have to be changed to a splitless model depending on the sensitivity of the particular MS being used. The retention time for LSD is concentration dependent. A series of standards of varying concentrations may have to be run in order to achieve the standard 0.04 minute retention time window.

5.2.0 TMS Derivative

At times, it may be necessary to derivatize weak LSD samples. The following is a summary of one possible method.

5.2.1 Reagents

Ammonium hydroxide (NH₄OH)

Methylene chloride, chloroform, or ethyl ether as solvents

MSTFA N-Methyl-N-trimethylsilyl-trifluoroacetamide

BSTFA bis(trimethylsilyl)trifluoroacetamide

5.2.2 Procedure

Place sample in concentrated NH₄OH and let soak for at least ten minutes. Add 200 ul of solvent and extract. Separate and evaporate the solvent. Add 30-200 ul of either MSTFA or BSTFA. Analyze on the GC/MS looking for the TMS derivative.

6.0.0 TLC Analysis

A T1 system followed by PDMAB color development works well for LSD. Other appropriate solvent systems, such as chloroform/methanol and acetone, may also be used. After the plate has been spotted with the sample extract, blank, and a standard, and the solvent has risen at least three quarters of the way up, remove the plate and dry. Check with UV and then develop with PDMAB. A purple color should develop with LSD.

6.1.0 The recipe for T1 is 7drops of ammonium hydroxide per 10ml of methanol.

6.2.0 The ratio of chloroform to methanol is 9/1.

6.3.0 PDMAB is 1gram of p'dimethylaminobenzaldehyde in 100ml of ethanol and 10ml of conc. hydrochloric acid.

7.0.0 Color Spot Tests

Marquis, grey color

Mandelin's, grey color

PDMAB, purple violet color

8.0.0 History

<u>Revision #</u>	<u>Issue or review date</u>	<u>History</u>	<u>Author or Reviewer</u>
0	11/02/01	Original Issue	D.C. Sincerbeaux
1	8/27/02	Scope & add #	D.C. Sincerbeaux
2	9/13/05	6.0.0 added blank	D.C. Sincerbeaux
3	1/12/07	Changed name, added pg #'s & history	D.C. Sincerbeaux
4	7/3/07	Added 3.5, 3.6 changed 3.4	D.C. Sincerbeaux
5	7/2/12	Changed 1.00, 3.2.0, 3.4.0	D.C. Sincerbeaux

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