

CASE REPORT

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The Isolation and Identification of Lysergic Acid Diethylamide (LSD) from Sugar Cubes and a Liquid Substrate

REFERENCE: Kilmer, S. D., "The Isolation and Identification of Lysergic Acid Diethylamide (LSD) from Sugar Cubes and a Liquid Substrate," *Journal of Forensic Sciences*, JFSCA, Vol. 39, No. 3, May 1994, pp. 860-862.

ABSTRACT: This report describes a simplified extraction technique for the analysis of LSD by GC/MS. The South Carolina Law Enforcement Division Forensic Laboratory recently received two suspected LSD cases involving four sugar cubes and seven food coloring bottles each containing a liquid substance. Following the extraction described in this report, both cases were subsequently confirmed by GC/MS and quantitated by HPLC.

KEYWORDS: toxicology, LSD

Lysergic acid diethylamide (LSD) is the hallucinogenic substance that is derived from the naturally occurring alkaloid lysergic acid. Lysergic acid is found in the ergot fungus (*Clavica purpurea*) which grows on certain rye plants [1]. LSD was first synthesized in 1938, while the psychotomimetic effects were discovered by accidental exposure in 1943 [2]. The U.S. Controlled Substance Act lists LSD as a Schedule I Substance. Schedule I substances are considered to have no accepted medical use and have a high potential for abuse.

The ingestion of LSD was a wide range of effects on the user. The primary effect on the user involves visual, as well as audio hallucinations. Distortion of time and space, vertigo, and nausea can also result depending on the quantity that is ingested [3].

Forensic laboratories encounter LSD in a variety of forms and substrates, such as "microdot" tablets, gelatin squares "windowpanes," or blotter paper squares. The most common form is blotter paper, which consists of perforated squares of paper that have been treated with a liquid containing LSD. While the concentration can vary, the average piece or "hit" of blotter paper contains 30 to 50 micrograms of LSD per square [2]. Aqueous solutions of LSD have also been applied to sugar cubes, postage stamps, as well as to food items such as crackers.

The South Carolina Law Enforcement Division (SLED) Drug Analysis Department

Received for publication 11 Feb. 1993; revised manuscript received 1 June, 8 Sept., 16 Nov. 1993; accepted for publication 18 Nov. 1993.

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receives an average of eight suspected LSD cases a month. While the most common form of LSD encountered at SLED is blotter paper, other forms such as microdots and postage stamps laced with LSD have also been analyzed. The average concentration of LSD in South Carolina is 25 to 50 micrograms per dosage unit.

Experimental

Laboratory procedure requires that a series of tests be conducted on a suspected LSD sample. First, a chemical spot test is performed using Erlich's test (p-dimethylamino-benzaldehyde or P-DMAB) [1]. Secondly, a visualization of the sample under both long and short wave ultraviolet light is a helpful step in the analysis due to the fluorescent properties of LSD [1]. A chemical extraction in methanol is followed by confirmation on the Hewlett-Packard 5970 gas chromatograph/mass spectrometer (GC/MS). The column is a 5% phenylmethyl silicone cross linked 25 meter \times 0.2 millimeter \times 0.33 micrometer GC column. The injection temperature is set at 270°C. The starting oven temperature is 100°C with a 24°C per minute temperature ramp up to 270°C. The method run time is 35 min with a sample retention time of 25 min. Quantitation of the sample is achieved on the Hewlett-Packard 1090 high performance liquid chromatograph (HPLC). The HPLC is equipped with the 1046A programmable fluorescence detector. The column is a Hewlett-Packard ODS Hypersil 5 μ m \times 100 mm \times 2.1 mm. The mobile phase used in the HPLC is 35/65 sodium phosphate buffer/methanol. The flow rate is 0.500 mL per minutes with a program time of 4 min.

Case Number 1

The SLED Drug Analysis Department received a case containing four sugar cubes that were believed to contain LSD. The P-DMAB chemical test resulted in a faint purple color. A positive screening is a dark purple color. Upon examination under the long and short wave UV light, all four cubes had regions, which brightly fluoresced. An extraction in methanol followed by an injection into the GC/MS was unsuccessful. An attempt to concentrate the sample down to a smaller volume and a reinjection into the GC/MS was also unsuccessful. In lieu of preparing a celite column for the separation of the LSD from the sugar substrate, a different chemical extraction method was successful. After the cube was crushed and placed into a beaker, several milliliters of 2N ammonium hydroxide (NH₄OH) were poured into the beaker covering the sugar. The NH₄OH was filtered into a test tube in which the filter paper and sugar were preserved for a future court exhibit. Methylene chloride was then added to the test tube using approximately twice the volume of the NH₄OH. The liquids were vortexed for approximately one minute after which the bottom organic layer was pipetted into a new test tube. At this step, attempts to inject a sample into the GC/MS were unsuccessful for the identification of LSD. The methylene chloride was allowed to evaporate under the fume hood to dryness. Approximately two or three milliliters of methanol were added to the test tube and vortexed for two minutes. A sample of methanol was injected into the GC/MS as a blank prior to the sample. Upon the injection of three microliters of the sample, the presence of LSD was confirmed. The sample was subsequently quantitated, as required by state law in South Carolina, using HPLC. Each sugar cube contained an average of 9 micrograms of LSD.

Case Number 2

A local police agency received information that a package was being sent through the U.S. Mail to a residence in Columbia, S.C. containing liquid LSD. Seven plastic food

coloring bottles were recovered and each sample contained an average volume of 3.2 mL of liquid. Upon performing the P-DMAB chemical test, all seven samples turned a dark purple color indicating a positive screening test of LSD. The liquid samples were extracted as described in case number 1 above using the 2N NH₄OH/methylene chloride/methanol extraction sequence. All seven samples were confirmed for the presence of LSD on the GC/MS and had an average quantitation of 744.1 µg per mL. Each vial could have been used to saturate approximately 47 squares of blotter paper at 50 µg per square. Therefore, if all seven vials had not been intercepted by the police, a total of 329 dosage units could have been created.

Conclusion

Forensic laboratories encounter many different exhibits of LSD in a variety of forms and substrates. While there are a variety of techniques available to isolate LSD, the base/methylene chloride/methanol sequence has been successful and reproducible in the isolation of LSD from different substrates. Further studies are currently being conducted at SLED with known standards of LSD and different laced substrates. The extraction sequence is being quantitated and further tested on such unique substrates.

References

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