ANALYSIS OF A CONTROLLED SUBSTANCE: LSD

Docoda Nunnery

Overview

- What is LSD?
- Drug Analysis
- Analyzing LSD
- Presumptive Testing
- Confirmatory Testing

What is LSD?- History

- LySergic acid Diethylamide
- Semi synthetic product of lysergic acid, a natural substance from Claviceps purpurea.
- Albert Hofmann 1938
- 1950s

”Model-Psychosis”
“Psychedelic Therapy”

Source 6

What is LSD?- Effects

- Hallucinogenic Effects
  - Altered state of consciousness
  - Illusions/ Hallucinations
  - Extreme Introspection
- Optimum dosage: 100-200µg
- High lasts: 6-10 hours (depending on the dose)

Source 6

What is LSD- Today

- A popular schedule 1 hallucinogenic drug.
- Illegally used worldwide
- *The National Survey on Drug Use and Health*
  - Major drug of abuse since 1970s

Source 6

What is LSD- Today

- Common to add drops of LSD solution into absorbent material.
- Most common: Sugar cubes, blotter paper, gelatin capsules. Tablets of various sizes, shapes, and colors
- “Window Panes” or “Pyramids”

Source 7

What is LSD- Today

Window Panes
(Gelatin Squares)

- Blotter Paper

Drug Analysis

- "One of the areas of forensic science where it is necessary to carry out an analytical investigation, to prove whether a controlled substance is present or otherwise."

- Types of analyses.
  - What is it?
  - How much?
  - Relationship?

Source 1

Drug Analysis

- Many analytical techniques, but ALL must adhere to the highest scientific standards

- SWGDRUG-> Scientific Working Group For The Analysis Of Drugs

Source 1 & 4

Drug Analysis- SWGDRUG

- The mission of SWGDRUG:

"To recommend minimum standards for the forensic examination of seized drugs and to seek their international acceptance."

Source 4

Drug Analysis- SWGDRUG

- SWGDRUG objectives:

1. Specify requirements for knowledge, skills, and abilities
2. Promote ethical standards and professional development
3. Provide minimum standards for examinations and reporting
4. Establishing quality assurance requirements.

Source 4

Drug Analysis- Instrumentation

- Based on discriminating power

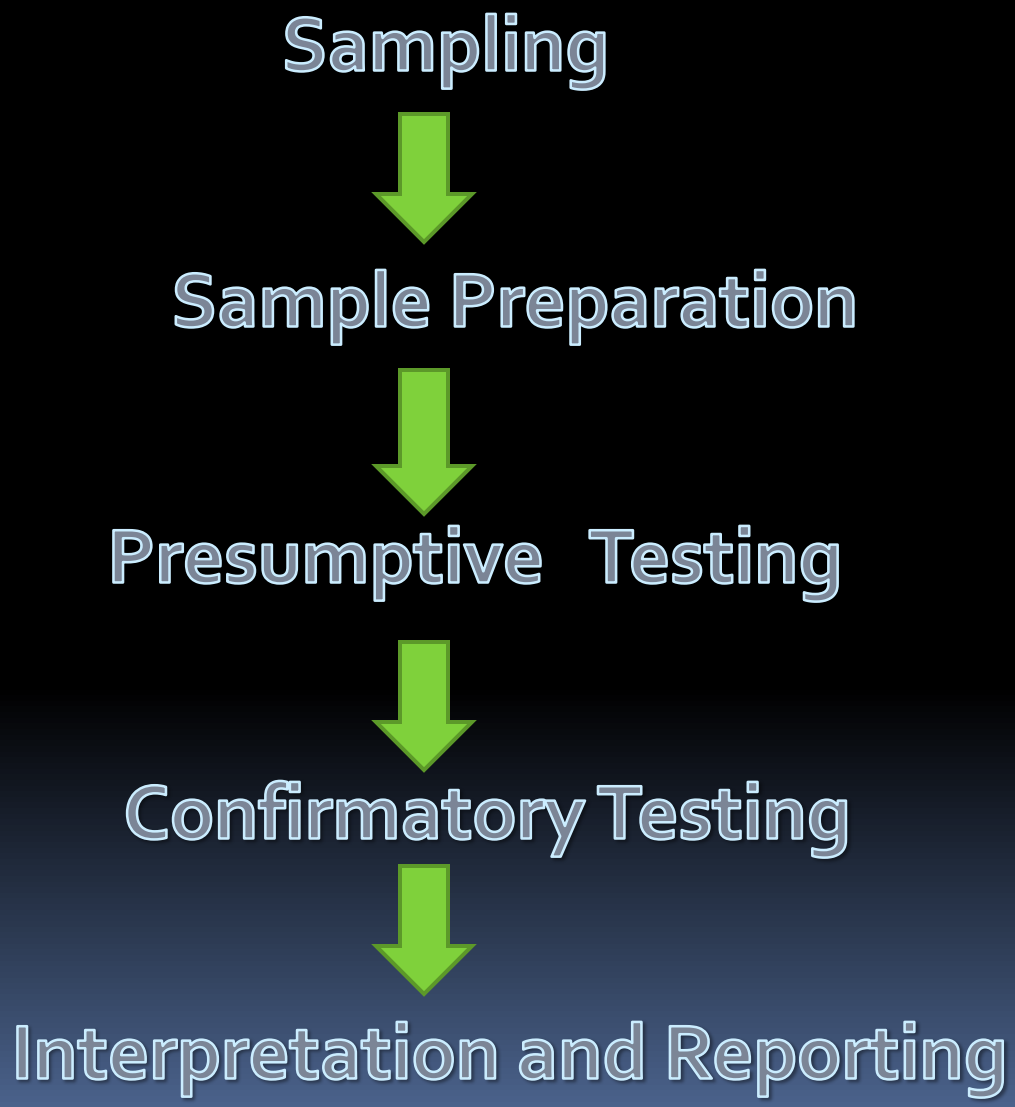
Category A	Category B	Category C
Infrared Spectroscopy	Capillary Electrophoresis	Color Tests
Mass Spectrometry	Gas Chromatography	Fluorescence Spectroscopy
Nuclear Magnetic Resonance Spectroscopy	Ion Mobility Spectrometry	Immunoassay
Raman Spectroscopy	Liquid Chromatography	Melting Point
	Microcrystalline Tests	Ultraviolet Spectroscopy
	Pharmaceutical Identifiers	
	Thin Layer Chromatography	
	Cannabis only: Macroscopic Examination Microscopic Examination	

Minimum Standards:

- Use Category A method: Incorporate → One other technique
- No Category A method: Incorporate → At least three techniques

Source 4

Drug Analysis- Workflow



Analyzing LSD- Sampling

- Vital that your sample be entirely representative of the bulk from which it was drawn.
- Different labs may have different sampling protocols.

Source 7

Analyzing LSD- Sampling

- Recommended Rule
  - Sample size up to 10-> Screen all
  - Sample size 11-27-> Screen Random ¾
  - Sample size 28+-> Screen ½
    - Minimum of 21, Maximum of 50.

- This is all to make sure that the entire population of samples are indeed LSD.

Source 7

Analyzing LSD- Sample Prep

- To prepare your sample for analysis by isolating the target compound (analyte)
- For LSD in blotter paper: Extract and filter.
- Two different extraction methods based on whether the analysis is qualitative or quantitative.

Source 7

Analyzing LSD- Sample Prep

Sample Preparation LSD

Qualitative

Extract with Methanol

Quantitative

Extract with 1% Tartaric Acid
Extract with Chloroform X3
Basify the aqueous layer
Extract with Chloroform X3
Recombine the Chloroform extracts
Filter or Centrifuge
Evaporate under nitrogen
Reconstitute in solvent.

Source 1,7

Analyzing LSD- Presumptive

- All ergot alkaloids will give similar results with these tests.
- Subject to false positives or false negatives.
  - Use of positive and negative controls
- Recommended Presumptive Tests
  - Fluorescence
  - Ehrlich Reagent
  - TLC

Source 1,7

Presumptive Testing: Fluorescence

- The extracted LSD is dropped onto filter paper and allowed to dry.
- The dried LSD is then subjected to long wavelength UV light (360nm).
- If LSD is present, blue fluorescence.
  - Positive control-> LSD
  - Negative control-> methanol

Source 1,7

Presumptive Testing: Ehrlich

- Simple color test
- Place LSD into a well plate
- Add a small amount of Ehrlich Reagent
- If positive-> A blue/purple color indicates LSD.
  - Positive control-> LSD
  - Negative control-> methanol

Source 1,7

Presumptive Test: Ehrlich

Source: www.chem.uwimona.edu.jm

Presumptive: TLC

- What is Chromatography?
  - Separation method
  - Two phases: Stationary phase and Mobile phase
  - Stationary Phase-> Usually solid
  - Mobile Phase-> A gas or liquid

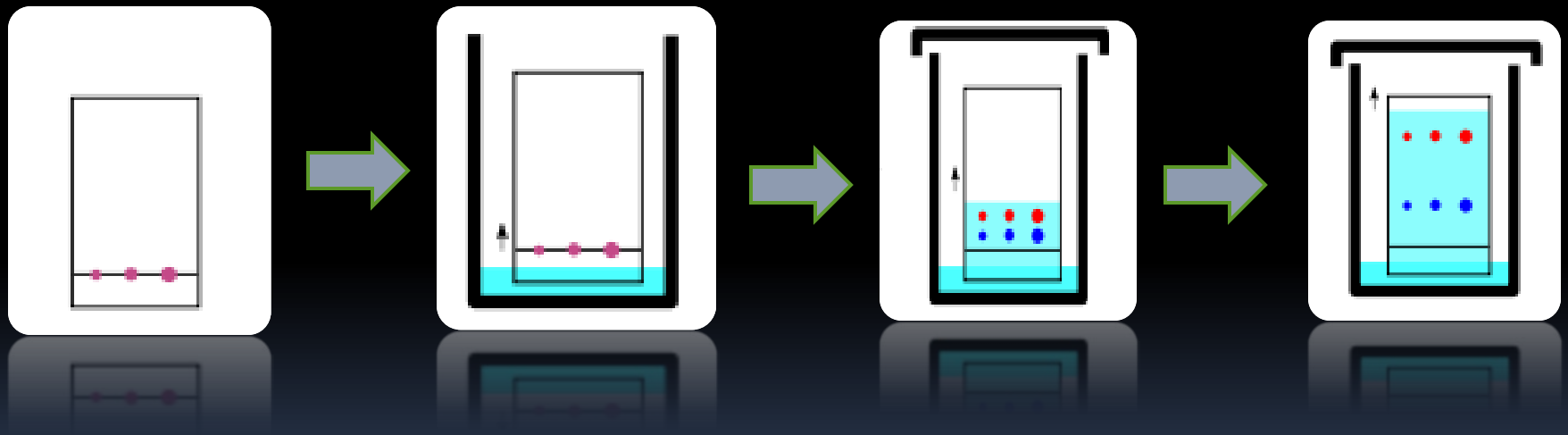
Presumptive: TLC

- In chromatography, a mixture separates because the components of that mixture have different affinities for the two phases (stationary/mobile).
- In other words, if a component was attracted to the mobile phase, it would move through the system faster than a component attracted to the stationary phase.

Source: 8

Presumptive: TLC

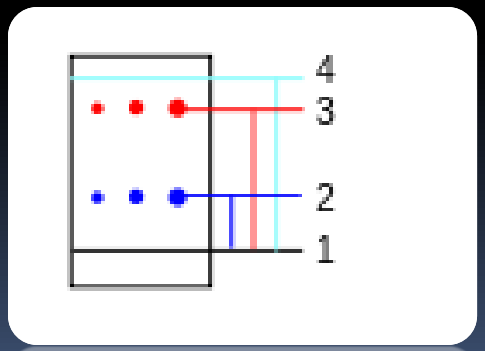
- TLC is a planar chromatography
  - Thin Layer Chromatography



Picture: Wikipedia.org

Presumptive: TLC

- Measure the Rf or Retardation factor: How far the compound moved.
- How far a compound moves in a specific solvent is a valuable characteristic.



Picture: Wikipedia.org

Source: 1,7

Presumptive: TLC

- Rapid, cost effective, although it cannot "prove" identity of LSD.
- Eliminates the samples that gave a positive color reaction but don't actually have LSD.
- Further eliminates a false positives, keeping an analyst from wasting valuable time and test materials.

Source: 1,7

Presumptive: TLC

- Procedure:
  - Silica gel plates, fluorescent dye
  - Solvent systems: Chloroform/methanol (9:1) and Chloroform/acetone (1:4)
  - After development, remove plates from tank.
  - Observe under 254nm and 360nm UV light.
  - Add Ehrlich Reagent-> Purple color
  - If same results as LSD (Rf, Color)-> Confirmatory Tests

Source: 1,7

Analyzing LSD- Confirmatory

- Better discriminating ability.
  - GC
  - HPLC
  - FTIR

Confirmatory: GC

- Gas Chromatography
  - Save chromatography concept except with a gas as the mobile phase and a column as the stationary phase.
  - Usually coupled with a Mass Spectrometer.
- Problematic- Why?
  - LSD is not very volatile.

Confirmatory: HPLC

- High Performance Liquid Chromatography
- Same basic principle of separation and Rf
- Uses high pressure to force solvent through closed column containing fine particles that give high resolution separations.

Confirmatory: HPLC

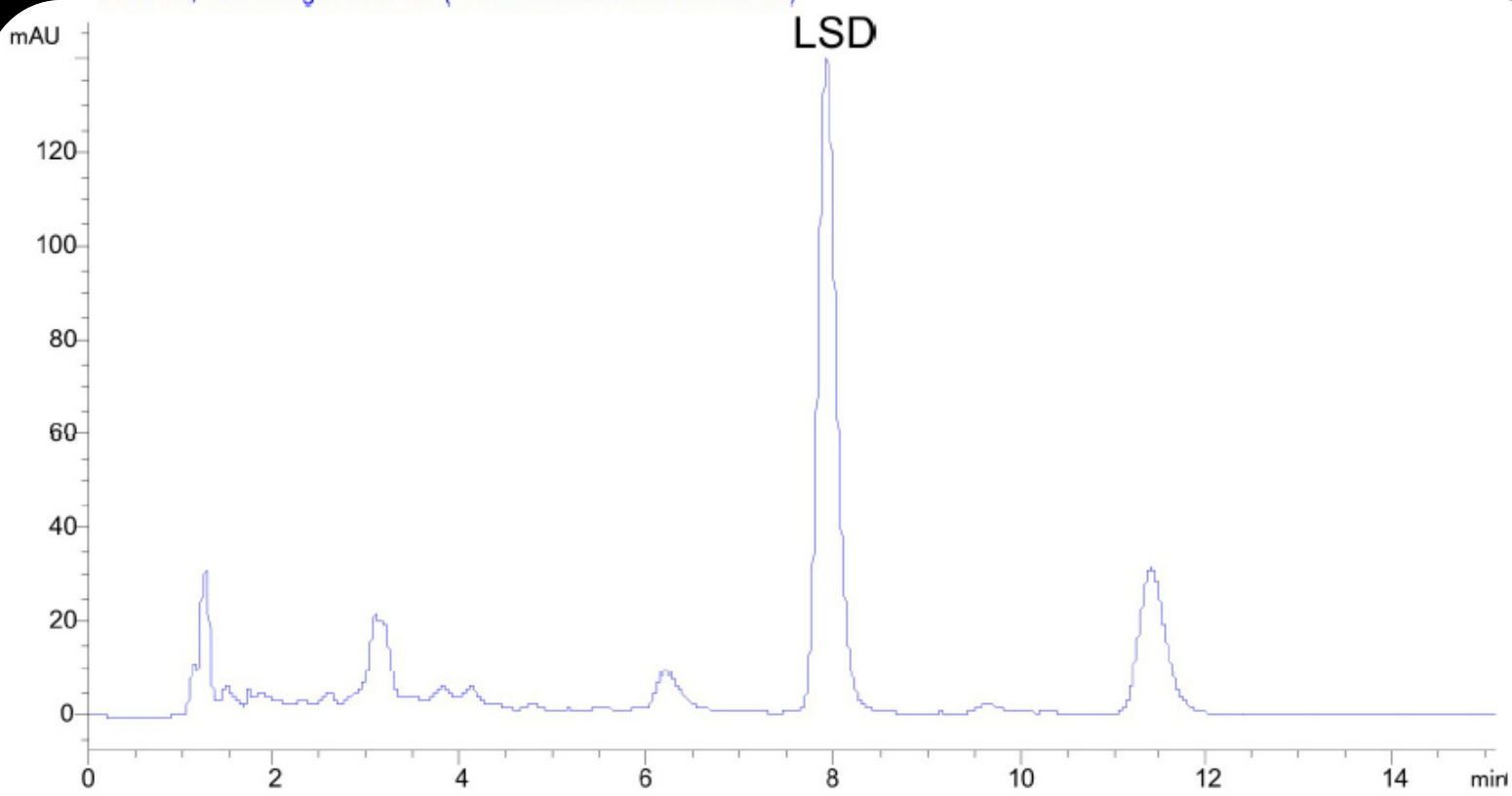
- With fluorescence detection, it's the most commonly used.
- Great resolution-> Good for quantifying

Source: 1,7

Confirmatory: HPLC

- LSD is particularly agreeable to fluorescence detection
- Both selectivity and sensitivity.
- Parameters:
  - Column: ODS silica
  - Solvent: Methanol (65%), 25 mM Na_2HPO_4, pH 8.0
  - Flow Rate: 1 mL/min
  - Detection: Fluorescence; Excitation 320 nm; Emission 400 nm.

Confirmatory: HPLC



Chromatogram after injection of blotter extract in methanol: water (1:1) tr LSD = 7.86 min.

Confirmatory: FTIR

- Fourier Transform Infrared Spectroscopy

Confirmatory: FTIR

- Used to measure the IR adsorption spectra of very small samples.
- Sample placed on a KBr disc and a microscope is then used to focus the IR beam onto the material.

Confirmatory: FTIR Procedure

- Blotter paper soaked in water (1 s)
- Excess water removed, placed on KBr disc
- Heated 1200 C for 1 min.
- Back to blotter: Dichloromethane/ammonia (100:1) -> removed via microsyringe-> KBr Disc.
- Spectrum recorded.

Confirmatory: FTIR

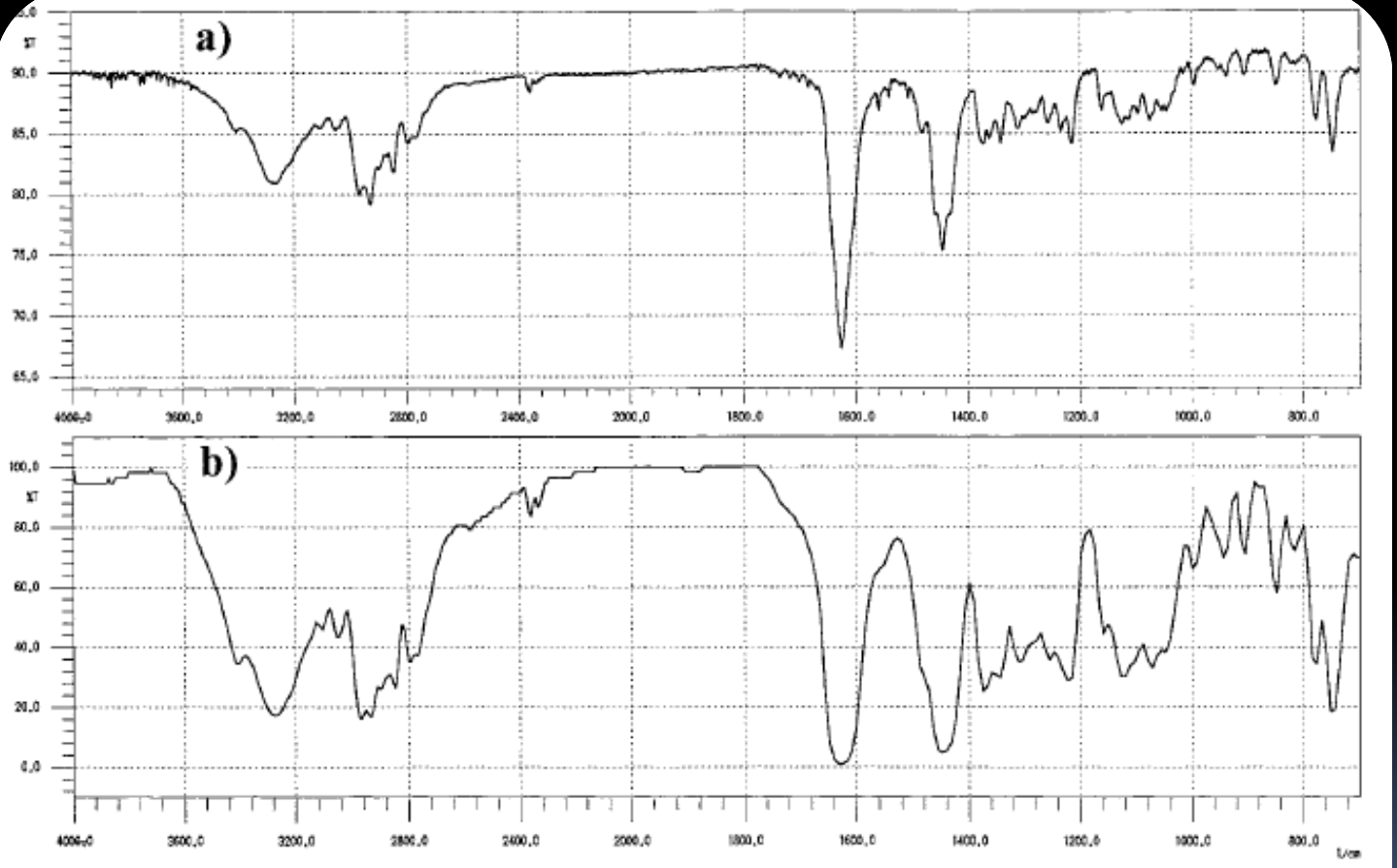


Fig. 1 FT-IR spectra of extract from the sample blotter paper and that of database. a) Sample blotter paper; b) database.

Conclusion

- LSD-> A hallucinogenic drug, that with a very small amount, can get you really high for a long time.
- Forms-> Blotter paper, tablets, gelatin square
- Analysis
  - Description
  - Extraction
  - Presumptive-> Ehrlich, Fluorescence, TLC
  - Confirmatory-> GC, HPLC, FTIR

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Acknowledgements

- Dr. Pamela Staton
- Dr. Lauren Waugh