#### 1. SYNONYMS

CFR: Lysergic acid diethylamide

**CAS #:** Base: 50-37-3

Other Names: D-lysergic acid diethylamide

N, N-diethyl-D-lysergamid Lysergsäure diethylamid

LAD LSD LSD-25

9,10-Didehydro-N,N diethyl-6-methylergoline-8-β-carboxamide

# Warning - LSD POWDER

LSD powder should be handled with the utmost care.

The analyst should work with an assistant using an empty hood where lights can be used to aid in UV light examination for LSD contamination. Once the container is opened, the chemist's hands and immediate area of the hood become contaminated. Anything needed outside the hood should be handled by the assistant. Dust mask, apron, and latex gloves should be worn. Dampening the gloves with a damp rag helps avoid problems associated with static electricity. Cloth gloves may be used over the latex gloves to minimize static as well. After analysis, the LSD container should be wiped off with a damp rag before replacing in the evidence bag. The chemist's gloves and the hood, including its contents should be decontaminated using diluted bleach. Decontamination is complete when all blue LSD fluorescence has been converted to yellow.

## 2. CHEMICAL AND PHYSICAL DATA

## 2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	C <sub>20</sub> H <sub>25</sub> N <sub>3</sub> O	323.4	80-85

Tartrate	C <sub>46</sub> H <sub>64</sub> N <sub>6</sub> O <sub>10</sub> *	861.0	198-200

## 2.2. SOLUBILITY

Form	A	С	E	Н	M	W
Base	VS	VS	VS	SS	VS	***
Tartrate	***	I	I	I	S	S

A = acetone, C = chloroform, E = ether, H = hexane, M = methanol and W = water, VS = very soluble, FS = freely soluble, S = soluble, PS = sparingly soluble, SS = slightly soluble, SS = very slightly soluble and SS = very slightly soluble

# 3. SCREENING TECHNIQUES

## 3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
<i>p</i> -Dimethylaminobenzaldehyde	Violet
UV light (long wave)	Blue fluorescence
UV light (short wave)	Blue fluorescence

For LSD dosage forms that contain dyes, the *p*-dimethylaminobenzaldehyde (PDMAB) color test should be done on a methanol extract of the sample. In a spot plate, add one drop of acidified PDMAB to the methanol extract and observe color. Alternatively for "blotters", a more sensitive method is to extract and concentrate the LSD with methanol followed by spotting onto a TLC plate. Allow it to dry then swab with PDMAB followed by exposure to hydrochloric acid fumes.

*Note*: Be aware that LSD will degrade with prolonged exposure to UV light. Many substances fluoresce blue with long wave or short wave UV light, but not both. Most types of paper fluoresce. Also, some colored paper and tablets absorb UV light so the blue fluorescence is not seen.

# 3.2. THIN-LAYER CHROMATOGRAPHY

## Visualization

UV light (long or short wave) Blue fluorescence

*p*-Dimethylamino-benzaldehyde Violet

Acidified iodoplatinate Black/purple

COMPOUND	RELATIV	RELATIVE R <sub>1</sub>		
	System TLC1	System TLC2		
LSD	1.0	1.0		
LSD (UV degradation)	0.2 1.2 1.4 1.6 1.8	***  ***  ***  ***		
LAMPA	0.8	0.9		
LAMPA (UV degradation)	0.2 1.1 1.3 1.5 1.8	***  ***  ***  ***		
iso-LSD	***	0.6		

# 3.3. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

# Method LSD-LCS1

Instrument: High performance liquid chromatograph equipped with diode array

Column: 5 μm Hypersil, 100 mm x 4.6 mm

Detector: UV, 310 nm

*Flow:* 1.2 mL/min

*Injection Volume:* 5.0 μL

Buffer: 2 mL concentrated ammonia, 400 mL methanol and 1598 mL

chloroform

Mobile Phase: Buffer: cyclohexane 30:70

Samples are to be dissolved in methanol and filtered with a 0.45-micron filter.

COMPOUND	RRT	COMPOUND	RRT
LSD	1.00	iso-LSD	1.42
LAMPA	1.00	ergonovine	3.71
ergotamine	1.34		

# 4. SEPARATION TECHNIQUES

Paper squares are extracted by soaking in dilute acid followed by basic extraction into methylene chloride. This provides LSD base for TLC, GC/MS, or Vapor Phase IR. Additional cleanup is needed for FT-IR as below. For fluorescence screening and color tests, extract the paper with methanol. For quantitation, soak the sample in methanol for at least four hours in the dark. Extracting overnight in the dark is preferred.

Tablets or capsules should be crushed then extracted similarly to the paper squares. Prior to the basic extraction, filter the acid extract to remove stearates and milk powder excipients. Dissolve gelatin squares in warm water before the acid-base extraction. Methanol may extract enough LSD from gelatin squares for fluorescence screening and color tests.

Another method is to use a micro-alumina column. Pre-rinse the column with methylene chloride then place the LSD base extract on the column. Elute the LSD off the column with a mixture of 5 drops methanol per 10 mL methylene chloride. Periodically, follow the LSD through the column with a fluorescent light. Change receivers just as the LSD comes off the micro-column. For samples containing iso-LSD, only collect the first two-thirds of the fluorescent band, because it closely follows behind LSD. Evaporate the solvent to produce residue suitable for FT-IR.

Separation of LSD from iso-LSD and trace excipients is also accomplished by developing a heavily spotted TLC plate. After developing, scrape off the streak and soak in dilute acid followed by basic extraction into methylene chloride. For paper not containing iso-LSD, basic extraction with hexane containing a small amount of chloroform may produce LSD pure enough for IR identification.

# 5. QUANTITATIVE PROCEDURES

# 5.1. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method LSD-LCQ1

## Standard Solution Preparation:

Accurately weigh and prepare a standard solution of lysergic acid diethylamide (LSD) tartrate at approximately 100 mg/mL using methanol.

# Sample Preparation:

Powder: Accurately weigh an amount of sample into a volumetric flask and dilute with methanol. If necessary, dilute the sample so the final concentration approximates the standard concentration. Filter sample with 0.45-micron filter. Take great caution in the amount of sample weighed due to its possible high purity. Blotter Paper: Place 5 to 10 squares (approximately " x " each) into a volumetric flask and add methanol. Allow the LSD to extract from the paper in the dark for at least four hours. If necessary, dilute the sample so the final concentration approximates the standard concentration. Filter sample with 0.45-micron filter.

*Instrument:* High performance liquid chromatograph equipped with diode array

Column: 5 μm Hypersil, 100 mm x 4.6 mm

Detector: UV, 310 nm

*Flow:* 1.2 mL/min

*Injection Volume:* 5.0 µL

Buffer: 2 mL concentrated ammonia, 400 mL methanol and 1598 mL

chloroform

Mobile Phase: Buffer: cyclohexane 30:70

Typical Retention Time: LSD: 4.0 min

*Linear Range:* 0.03 - 2.0 mg/mL

**Repeatability:** RSD less than 0.6%

Correlation Coefficient: 0.999

Accuracy: Error less than 5%

# 6. QUALITATIVE DATA

#### 6.1. ULTRAVIOLET SPECTROPHOTOMETRY

LSD Tartrate at 40 µg/mL

SOLVENT	MAXIMUM ABSORBANCE
	(NM)

Methanol	310
Sulfuric acid	310
Ammonium hydroxide	308

See spectra on the following pages for FT-IR, Mass Spectrometry, Nuclear Magnetic Resonance, and Vapor Phase IR.

#### 7. REFERENCES

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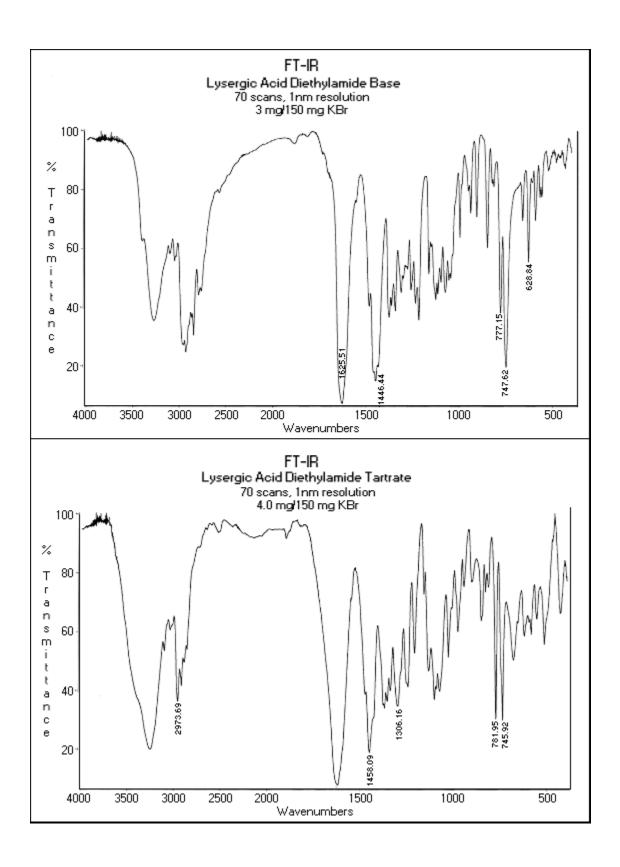
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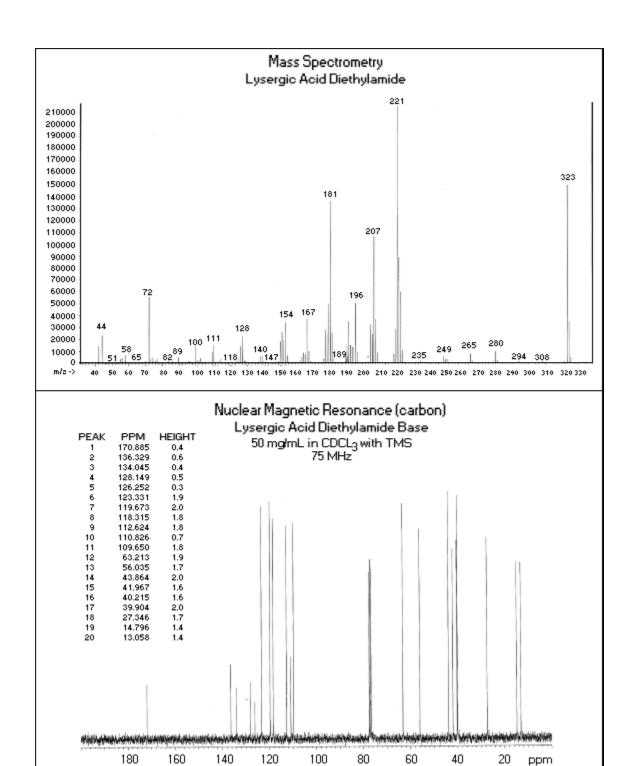
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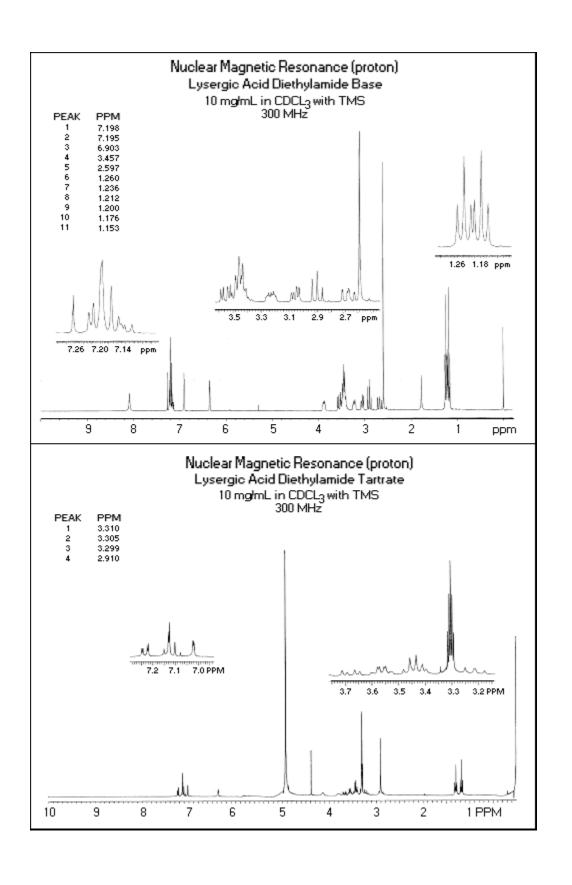
# 8. ADDITIONAL RESOURCES

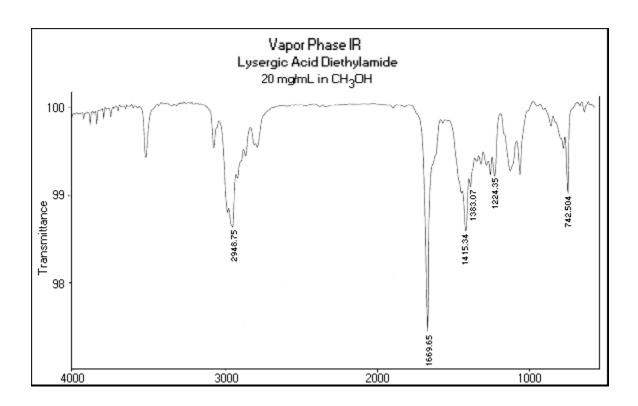
**Forendex** 

Wikipedia









\* Solvated with methanol ( $C_{20}H_{25}N_3O)_2$   $C_4H_6O_6$  2  $CH_3OH$  \*\*\* No data available